Supporting Information

A Convergent Paired Electrochemical Strategy for the Decarboxylative C(sp^2)-C(sp^3) Bond Formation

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General Information

NMR spectra were recorded on Varian 400 MHz and Bruker AV500 instruments at ambient temperature with $CDCl_3$ as the solvent unless otherwise stated. Chemical shifts are reported in parts per million relative to $CDCl_3$ (^1H, δ 7.26 for $CDCl_3$; ^{13}C, δ 77.16 for $CDCl_3$ unless otherwise stated). Data for ^1H NMR are reported as follows: chemical shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad), coupling constants (Hz), and integration. High resolution mass spectra (HRMS) were recorded at NIBS Metabolomics Center using an Thermo Scientific Q Exactive HF-X Hybrid Quadrupole-Orbitrap MS. Analytical thin layer chromatography was performed using 0.25 mm silica gel 60-F plates. Column chromatography was performed using 300-400 mesh silica gel. Yields refer to chromatographically and spectroscopically pure materials, unless otherwise stated. All reactions were carried out under an inert argon atmosphere with dry solvents under anhydrous conditions unless otherwise stated. All glassware was dried in a drying oven before using. Anhydrous NMP (*N*-methyl-2-pyrrolidone), DMF (*N, N*-dimethylformamide), and DMA (*N, N*-dimethylacetamide) were purchased from ACROS or Sigma-Aldrich®.

General Procedure

Part I. Preparation of NiBr$_2$•glyme•2bpy or NiBr$_2$•glyme•2dtbbpy solution (0.01 M based on Ni)

A screw-capped culture tube charged with a magnetic stir bar was taken into a glove box. NiBr$_2$•glyme (6.2 mg, 0.02 mmol), bpy (6.25 mg, 0.04 mmol) or dtbbpy (10.74 mg, 0.04mmol), and anhydrous NMP (2 mL) were added. The resulting mixture was stirred at 80 °C until a clear green solution was afforded (usually 2 h). (*Note 1*).

Part II. Electrochemical decarboxylative cross-coupling (*Note 2*)

A reaction tube charged with a magnetic stir bar was moved into a glove box. NaI (36 mg, 0.24 mmol, 1.2 equiv), PPh$_3$ (157 mg, 0.6 mmol, 3.0 equiv), NHPI (49 mg, 0.3 mmol, 1.5 equiv), DMAP (36.7 mg, 0.3 mmol, 1.5 equiv), aryl iodide or vinyl iodide (0.3 mmol, 1.5 equiv), the carboxylic acid (0.2 mmol, 1.0 equiv), and anhydrous NMP (2 mL) were added. The resulting mixture was stirred until all reactants were completely dissolved (*ca.* 5 minutes). The NiBr$_2$• glyme•2bpy solution (2 mL) or the NiBr$_2$•glyme•2dtbbpy solution (2 mL) was added before electrodes [(+)-graphite|(–)-Ni foam] (*Note 3*) were installed, and the reaction mixture was stirred under a constant current of 3 mA for 12 hours.

After completion, the electrolytic cell was moved from the glove box, and electrodes were rinsed with EtOAc (7 mL). The crude reaction mixture was further diluted with EtOAc (30 mL) (*Note 4*). The resulting mixture was washed with water (40×2 mL) and brine (40 mL) sequentially whereby the aqueous layers were back-extracted with EtOAc (1×40 mL). The combined organic layers were dried over Na$_2$SO$_4$ and concentrated under reduced pressure. Purification by column chromatography followed by further purification by preparative TLC or preparative HPLC afforded the desired products.

Note 1: Freshly prepared NiBr$_2$•glyme•2bpy solution or NiBr$_2$•glyme•2dtbbpy solution provides the best yield of coupling products.

Note 2: This decarboxylative arylation is recommended to be operated in the glove box under N$_2$ or Ar. If the reaction was conducted out of the glove box, the follow procedure was followed: To a reaction tube charged with a magnetic stir bar was added NaI (36 mg, 0.24 mmol, 1.2 equiv), PPh$_3$ (157 mg, 0.6 mmol, 3.0 equiv), NHPI (49 mg, 0.3 mmol, 1.5 equiv), DMAP (36.7 mg, 0.3 mmol, 1.5 equiv), aryl iodide or vinyl iodide (0.3 mmol, 1.5 equiv, *if solid*), and the carboxylic acid (0.2 mmol, 1.0 equiv, *if solid*). The reaction tube was covered with the assembled electrodes and sealed with electrical tape. The assembled electrolytic cell was evacuated and backfilled with argon for three times before an argon balloon was added. NMP (2 mL) was added, and the mixture was stirred until all reactants were completely dissolved before the addition of the solution of NiBr$_2$•glyme•2bpy, aryl iodide or vinyl iodide (0.3 mmol, 1.5 equiv, *if liquid*), and the carboxylic acid (0.2 mmol, 1.0 equiv, *if liquid*). The reaction mixture was stirred under a constant current of 3 mA for 12 hours.

Note 3: Low density graphite was used as anode.

Note 4: If the product is not sensitive to H_2O_2, a few drops of aq. H_2O_2 could be added after the completion of the reaction to help remove the excess PPh_3.

Graphical Supporting Information

I. Procedure for the preparation of $NiBr_2$•glyme•2bpy or $NiBr_2$•glyme•2dtbbpy solution (0.01 M based on Ni).

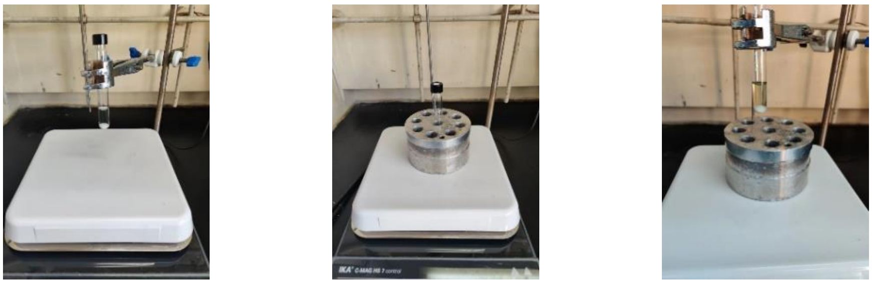


Figure S1. *Left:* A screw-capped culture tube was charged with $NiBr_2$•glyme and bpy; *Middle:* The suspension of $NiBr_2$•glyme and bpy in NMP was heated to 80 °C; *Right:* After 2 h stirring, the solution was cooled to room temperature.

II. Assembling of the electrolytic cell.

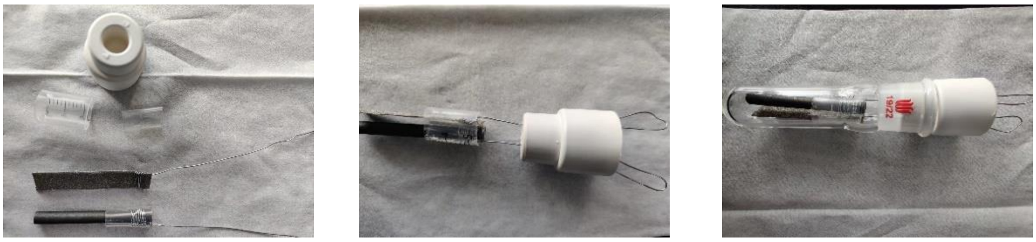


Figure S2. *Left:* Materials for making the electrodes; *Middle:* The assembled electrodes; *Right:* The assembled electrolytic cell.

III. Electrochemical decarboxylative cross-coupling.

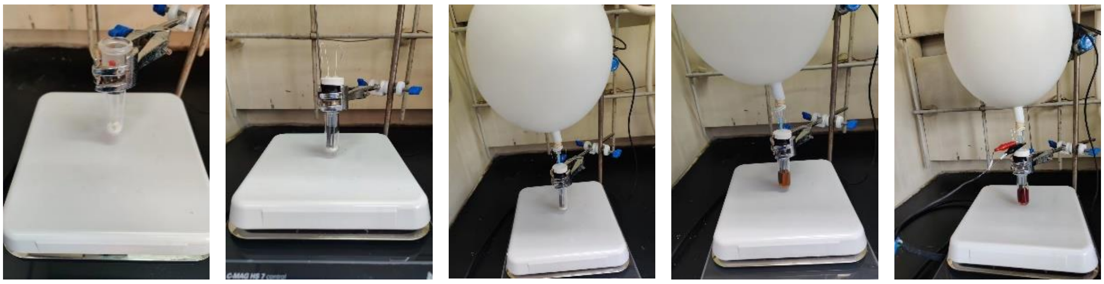
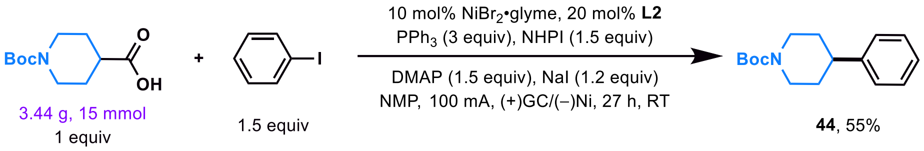


Figure S3. From left to right: (1) A reaction tube was charged with NaI, PPh_3, DMAP, NHPI and solid acid; (2) The reaction tube was covered with the assembled electrodes and sealed with electrical tape before being evacuated and backfilled with argon (three times); (3) A argon balloon was added; (4) NMP was added, and the mixture was stirred until all reactants were completely dissolved before the addition of solution of $NiBr_2$•glyme•2bpy and PhI. The resulting mixture was stirred until the reaction solution become homogeneous; (5) Starting the reaction with an electrochemical equipment with a constant current of 3 mA (ca. 10 mm electrodes were immerged into the reaction solution).

IV. Gram-scale electrochemical decarboxylative cross-coupling.



A 250 mL round bottom flask charged with a magnetic stir bar was taken into a glove box. $NiBr_2$•glyme (463 mg, 1.5 mmol), dtbbpy (805.2 mg, 3.0 mmol), and anhydrous NMP (150 mL) were added. The resulting mixture was stirred at 80 °C until a clear green solution was afforded (usually 2 h).

N-Boc-piperidine-4-carboxylic acid (3.44 g, 15 mmol, 1.0 equiv), NaI (2.7 g, 18 mmol, 1.2 equiv), PPh_3 (11.8 g, 45 mmol, 3.0 equiv), NHPI (3.67 g, 22.5 mmol, 1.5 equiv), DMAP (2.75 g, 22.5 mmol, 1.5 equiv) were added to the custom-made reaction flask (as shown in the following Figure). The reaction flask was assembled with in-house made electrodes (as shown below). The assembled flask was evacuated and backfilled three times with argon before the addition of anhydrous NMP (150 mL). The resulting mixture was stirred until all reactants were completely dissolved (*ca.* 5 minutes). Iodobenzene (4.59 g, 22.5 mmol, 1.5 equiv) and $NiBr_2$•glyme•2dtbbpy solution (150 mL) was added, and the reaction mixture was stirred under a constant current of 100 mA for 27 hours.

After completion, the reaction mixture was diluted with EtOAc (300 mL), and washed with H_2O (2×300 mL), brine (1×300 mL). The organic layer was separated, and the aqueous phases were back-extracted with EtOAc (2×300 mL). The combined organic phases were then dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. Purification by flash chromatography on silica gel (9:1, Petroleum ether: EtOAc) afforded the product **44** (2.15 g, 55%).

Figure S4. From left to right: (1) The electrode fabrication using graphite flakes and nickel foam. (2) To the custom-made reaction flask was added NaI, PPh_3, DMAP, NHPI, and *N*-Boc-piperidine-4-carboxylic acid; (3) The sealing of the flask by covering the flange and inserting the rubber stopper securely; (4) The reaction flask undergoes three cycles of evacuation and backfilling with argon to create an inert atmosphere; NMP (*N*-Methyl-2-pyrrolidone) is added to the evacuated and argon-filled reaction flask. The mixture is stirred until complete dissolution of all reactants is achieved. Subsequently, a solution containing $NiBr_2$•glyme•2dtbbpy and PhI is introduced to the reaction mixture. (5) The reaction mixture was stirred for a few more minutes to make the reaction solution homogeneous before starting the reaction on an electrochemical equipment with a constant current.

Mechanistic Studies

I. Relative reactivity study

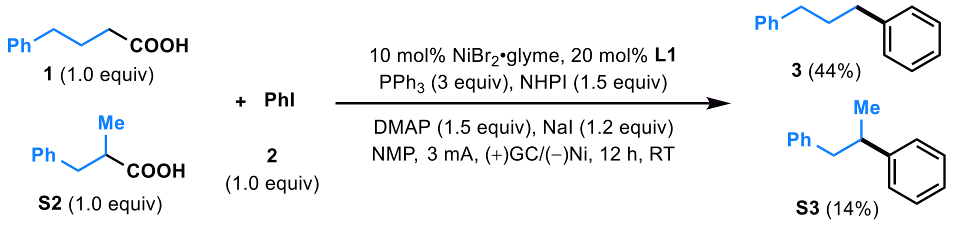
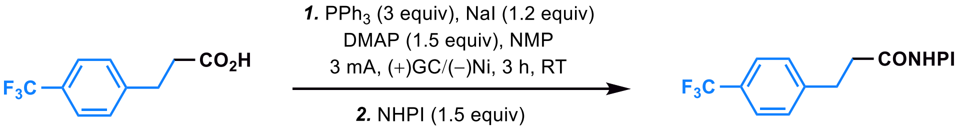


Figure S5. Competitive experiment of different carboxylic acids.

Procedure: On 0.2 mmol scale, **General Procedure** was followed with **1** (1.0 equiv), **S2** (1.0 equiv) and iodobenzene (1.0 equiv). Purification by column chromatography (silica gel, Petroleum Ether) followed by preparative TLC (silica gel, 20:1, Petroleum ether: EtOAc) afforded a mixture of **3** and **S3**. The yields of **3** and **S3** was determined by ^{1}H NMR with CH_2BrI (0.1 mmol) as internal standard.

II. Reaction monitoring by ^{19}F NMR[1-2]



65 **67**

Procedure: A reaction tube charged with a magnetic stir bar was moved into a glove box. NaI (36 mg, 0.24 mmol, 1.2 equiv), PPh_3 (157 mg, 0.6 mmol, 3.0 equiv), DMAP (36.7 mg, 0.3 mmol, 1.5 equiv), the carboxylic acid **65** (0.2 mmol, 1.0 equiv), and anhydrous NMP (4 mL) were added. The resulting mixture was stirred until all reactants were completely dissolved (*ca.* 5 minutes). The assembled electrodes [(+)-graphite|(−)-Ni foam] were installed, and the reaction mixture was stirred under a constant current of 3 mA. After stirring for 3 h, the electric current was ceased, and NHPI (49 mg, 0.3 mmol, 1.5 equiv) was added. The reaction mixture was stirred for 3h before being detected by ^{19}F NMR.

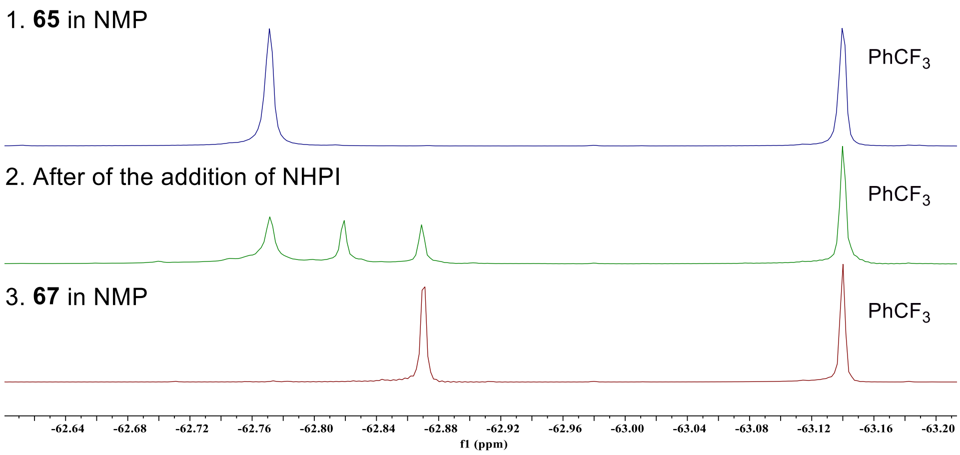


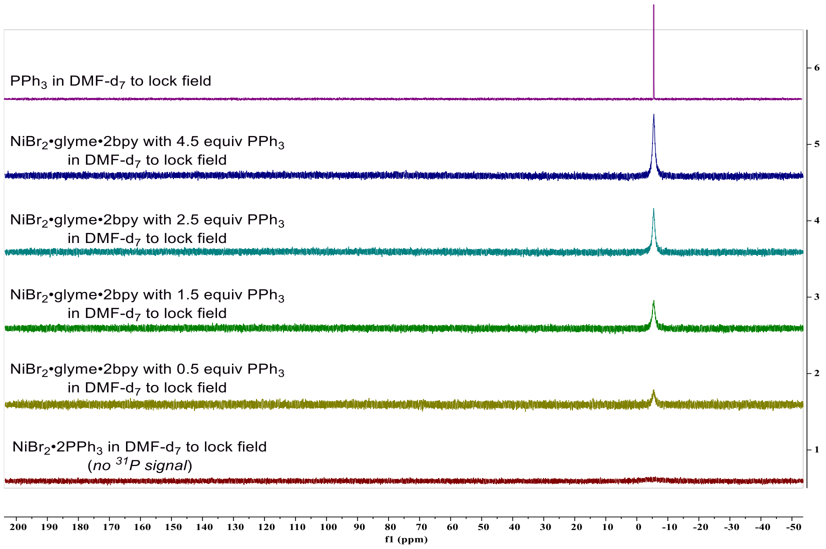
Figure S6. Tracking the generation of NHPI ester of **65** by ^{19}F NMR. (1) ^{19}F NMR of the solution of **65** (1 equiv), NaI (1.2 equiv), PPh$_3$ (3 equiv), and DMAP (1.5 equiv) in NMP; (2) ^{19}F NMR of the reaction mixture; (3) ^{19}F NMR of the solution of RAE **67** in NMP.

III. Ligand exchange experiments

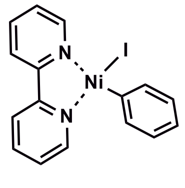
The following NMR experiment excluded the possibility that PPh$_3$ worked as a ligand of Ni catalyst.

$$\underset{(1\ \text{equiv})}{\mathrm{NiBr_2{\bullet}glyme}} + \underset{(2\ \text{equiv})}{\mathrm{bpy}} \xrightarrow[80\ °\mathrm{C},\ 2\ \mathrm{h}]{\mathrm{DMF}\text{-}d_7} \mathrm{NiBr_2{\bullet}glyme{\bullet}2bpy} \xrightarrow[\text{r.t., 2 h}]{\substack{\mathrm{PPh_3} \\ (0.5\ \text{to}\ 4.5\ \text{equiv})}} \text{no ligand exchange}$$

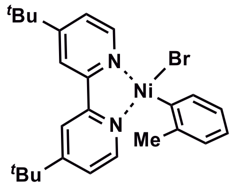
A screw-capped culture tube charged with a magnetic stir bar was moved into a glove box. NiBr$_2$•glyme (30.9 mg, 0.1 mmol), bpy (**L1**, 31.2 mg, 0.2 mmol), and DMF-d_7 (1 mL) were added. The resulting mixture was stirred at 80 °C for 2 hours and then cooled to room temperature. PPh$_3$ was added, and the mixture was stirred for additional 2 hours at room temperature. The reaction mixture was detected by ^{31}P NMR. ^{31}P NMR (162 MHz, DMF-d_7) δ –5.49 (PPh$_3$). It was shown that the addition of PPh$_3$ didn't result in the ligand exchange.



IV. SWV studies of aryl-Ni(II) complex



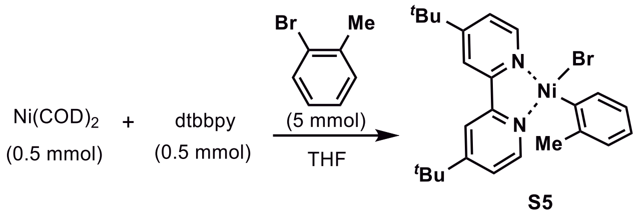
S4



S5

Initially, we endeavored to synthesize the aryl-Ni(II) complex **S4** to assess its reduction potential through SWV/CV. Regrettably, **S4** was deemed too unstable for characterization. Consequently, our focus shifted to the preparation of **S5**, a known and relatively stable compound,[5] to investigate the reduction potential of the aryl-Ni(II) complex.

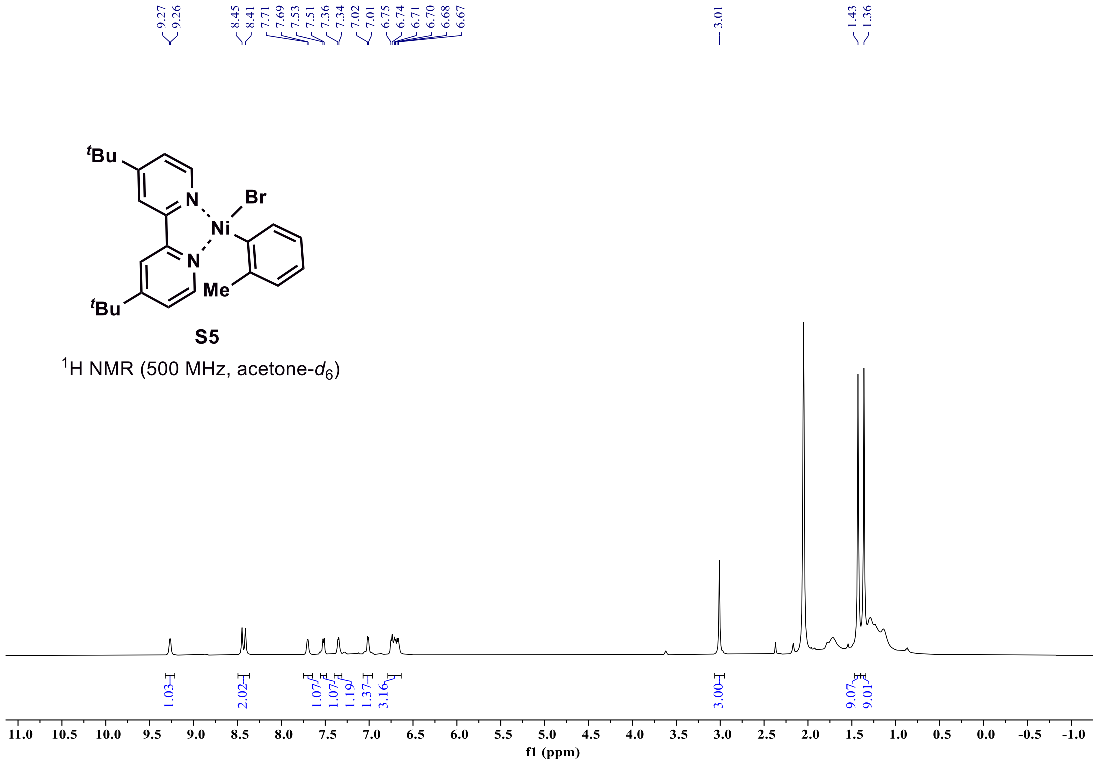
(2) Synthesis of [(dtbbpy)Ni(II)(2-tolyl)Br] (S5)



A screw-capped culture tube charged with a magnetic stir bar was moved into a glove box. $Ni(COD)_2$ (0.5 mmol, 136 mg), dtbbpy (0.5 mmol, 140 mg), and dry THF (4 mL) were added. The resulting deep purple mixture was stirred for 2 hours. 2-Bromotoluene (5 mmol, 850 mg) was added and stirred for additional 3 hours. Dry *n*-pentane (20 mL) was added to the deep red colored mixture and filtered. The residue was washed with *n*-pentane (5 x10 mL) and dried under vacuum to give the desired complex as an orange powder (181.6 mg, 73%).

^{1}H NMR (500 MHz, Acetone-d_6): δ 9.27 (d, J = 5.7 Hz, 1H), 8.43 (d, J = 19.4 Hz, 2H), 7.70 (d, J = 5.7 Hz, 1H), 7.52 (d, J = 7.2 Hz, 1H), 7.35 (d, J = 6.3 Hz, 1H), 7.01 (d, J = 6.1 Hz, 1H), 6.77 – 6.65 (m, 3H), 3.01 (s, 3H), 1.43 (s, 9H), 1.36 (s, 9H).

Spectral data matched those previously reported.[5]

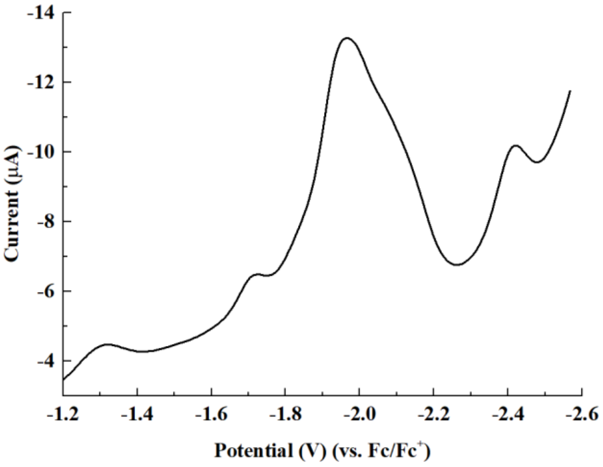


S5

^{1}H NMR (500 MHz, acetone-d_6)

(3) SWV studies of [(dtbbpy)Ni(II)(2-tolyl)Br] (S5)

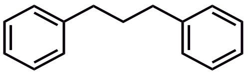
Square wave voltammetry experiments were performed from positive to negative potential with an amplitude of 25 mV, frequency of 40 Hz, and a step potential of 5 mV, by using a glassy carbon working electrode, a platinum wire counter electrode, and an Ag/AgNO$_3$ (10 mM with 0.1 M LiClO$_4$ in NMP) reference electrode. All the potentials values reported in this work have been measured vs. Ag/AgNO$_3$ reference electrode and converted to Fc/Fc$^+$ by adding ferrocene/ ferrocenium as internal standard (E$_{1/2}$(Fc/Fc$^+$) ≈ 0.07 V vs. Ag/AgNO$_3$) at the end of each experiment. All the electrochemical experiments were carried out in NMP with the presence of 0.1 M LiClO$_4$ as supporting electrolyte under argon at 25 ºC.



The resulting SWV indicated that the reduction potential of (dtbbpy)Ni(II)(2-tolyl)Br **(S5)** is –1.97 V [to Ni(I) species], which was similar to previous reports.[6]

Characterization Data

Decarboxylative arylation



3

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **3** (34.0 mg, 87%).

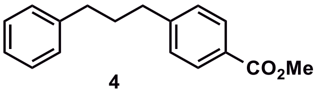
Physical state: colorless oil;

TLC: R_f = 0.76 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.26 (m, 4H), 7.22 – 7.16 (m, 6H), 2.71 – 2.60 (m, 4H), 2.03 – 1.92 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 142.4, 128.6, 128.4, 125.9, 35.6, 33.1;

HRMS (ESI): Calcd for $C_{15}H_{17}$ $[M+H]^+$: 197.1325; found 197.1330.



4

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), methyl 4-iodobenzoate (78.6 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **4** (39.5 mg, 78%).

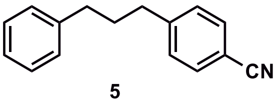
Physical state: colorless oil;

TLC: R_f = 0.40 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 – 7.91 (m, 2H), 7.31 – 7.22 (m, 4H), 7.22 – 7.16 (m, 3H), 3.90 (s, 3H), 2.72 – 2.60 (m, 4H), 2.02 – 1.91 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.3, 148.0, 142.0, 129.8, 128.6, 128.6, 128.5, 127.9, 126.0, 52.1, 35.5, 35.5, 32.7;

HRMS (ESI): Calcd for $C_{17}H_{19}O_2$ $[M+H]^+$: 255.1380; found 255.1385.



5

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 4-iodobenzonitrile (68.7 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **5** (35.6 mg, 80%).

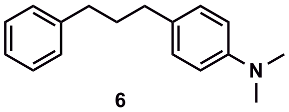
Physical state: colorless oil;

TLC: R_f = 0.34 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 – 7.54 (m, 2H), 7.32 – 7.25 (m, 4H), 7.23 – 7.15 (m, 3H), 2.73 – 2.62 (m, 4H), 2.01 – 1.92 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 148.1, 141.7, 132.3, 129.3, 128.5, 128.5, 126.1, 119.3, 109.8, 35.6, 35.4, 32.5;

HRMS (ESI): Calcd for $C_{16}H_{16}N$ $[M+H]^+$: 222.1277; found 222.1283.



6

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 4-iodo-*N*, *N*-dimethyl-benzenamine (74.1 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **6** (34.8 mg, 73%).

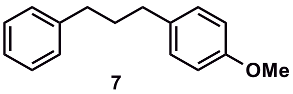
Physical state: colorless oil;

TLC: R_f = 0.42 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.27 (m, 2H), 7.22 – 7.17 (m, 3H), 7.11 – 7.07 (m, 2H), 6.74 – 6.71 (m, 2H), 2.92 (d, *J* = 0.8 Hz, 6H), 2.68 – 2.63 (m, 2H), 2.61 – 2.56 (m, 2H), 1.99 – 1.90 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 149.1, 142.7, 130.6, 129.1, 128.6, 128.4, 125.7, 113.1, 41.1, 35.6, 34.5, 33.4;

HRMS (ESI): Calcd for $C_{17}H_{22}N$ $[M+H]^+$: 240.1747; found 240.1752.



7

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 4-iodoanisole (70.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **7** (25.3 mg, 56%).

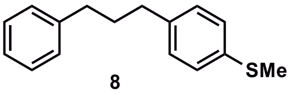
Physical state: colorless oil;

TLC: R_f = 0.54 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.25 (m, 2H), 7.21 – 7.16 (m, 3H), 7.12 – 7.09 (m, 2H), 6.87 – 6.77 (m, 2H), 3.80 (s, 3H), 2.64 (t, *J* = 7.7 Hz, 2H), 2.60 (t, *J* = 7.7 Hz, 2H), 1.98 – 1.87 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 157.8, 142.5, 134.5, 129.4, 128.6, 128.4, 125.8, 113.8, 55.4, 35.5, 34.6, 33.3;

HRMS (ESI): Calcd for $C_{16}H_{19}O$ $[M+H]^+$: 227.1430; found 227.1436.



8

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 4-iodothioanisole (75.0 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **8** (34.7 mg, 72%).

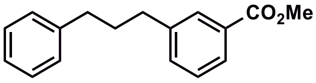
Physical state: colorless oil;

TLC: R$_f$ = 0.62 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.26 (m, 2H), 7.23 – 7.17 (m, 5H), 7.14 – 7.09 (m, 2H), 2.67 – 2.59 (m, 4H), 2.47 (s, 3H), 1.99 – 1.90 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 142.3, 139.6, 135.3, 129.1, 128.6, 128.4, 127.3, 125.9, 35.5, 35.0, 33.0, 16.5;

HRMS (ESI): Calcd for $C_{16}H_{19}S$ $[M+H]^+$: 243.1202; found 243.1207.



9

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), methyl 3-iodobenzoate (78.6 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **9** (36 mg, 71%).

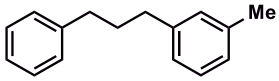
Physical state: colorless oil;

TLC: R$_f$ = 0.40 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 – 7.81 (m, 2H), 7.40 – 7.26 (m, 4H), 7.22 – 7.16 (m, 3H), 3.91 (s, 3H), 2.71 (t, J = 7.6 Hz, 2H), 2.66 (t, J = 7.6 Hz, 2H), 2.06 – 1.92 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.4, 142.7, 142.1, 133.3, 130.3, 129.7, 128.6, 128.5, 127.2, 126.0, 52.2, 35.5, 35.3, 33.0;

HRMS (ESI): Calcd for $C_{17}H_{19}O_2$ $[M+H]^+$: 255.1380; found 255.1385.



10

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 3-iodotoluene (65.4 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **10** (35 mg, 83%).

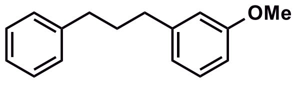
Physical state: colorless oil;

TLC: R$_f$ = 0.73 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.25 (m, 2H), 7.21 – 7.15 (m, 4H), 7.08 – 6.93 (m, 3H), 2.68 – 2.58 (m, 4H), 2.33 (s, 3H), 2.01 – 1.82 (m, 2H);

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 142.5, 142.4, 138.0, 129.4, 128.6, 128.4, 128.3, 126.6, 125.9, 125.6, 35.7, 35.5, 33.1, 21.6;

HRMS (ESI): Calcd for $C_{16}H_{19}$ [M+H]$^+$: 211.1481; found 211.1487.



11

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 3-iodoanisole (70.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **11** (37.8 mg, 84%).

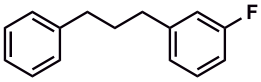
Physical state: colorless oil;

TLC: R$_f$ = 0.67 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.32 – 7.26 (m, 2H), 7.23 – 7.16 (m, 4H), 6.82 – 6.72 (m, 3H), 3.80 (s, 3H), 2.69 – 2.61 (m, 4H), 2.02 – 1.92 (m, 2H);

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 159.7, 144.0, 142.4, 129.4, 128.6, 128.4, 125.9, 121.0, 114.3, 111.1, 55.2, 35.6, 35.5, 32.9;

HRMS (ESI): Calcd for $C_{16}H_{19}O$ [M+H]$^+$: 227.1430; found 227.1436.



12

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 1-fluoro-3-iodobenzene (66.6 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **12** (29.4 mg, 69%).

Physical state: colorless oil;

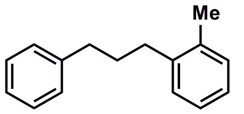
TLC: R$_f$ = 0.78 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.32 – 7.25 (m, 2H), 7.25 – 7.16 (m, 4H), 6.97 – 6.94 (m, 1H), 6.91 – 6.85 (m, 2H), 2.68 – 2.61 (m, 4H), 2.02 – 1.91 (m, 2H);

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 163.1 (d, *J* = 244.9 Hz), 145.0 (d, *J* = 7.4 Hz), 142.1, 129.8 (d, *J* = 8.6 Hz), 128.6, 128.5, 126.0, 124.2 (d, *J* = 2.7 Hz), 115.4 (d, *J* = 20.6 Hz), 112.8 (d, *J* = 20.9 Hz), 35.4, 35.3, 32.8;

^{19}F NMR (376 MHz, CDCl$_3$): *δ* –113.95 – –114.00 (m);

HRMS (ESI): Calcd for $C_{15}H_{16}F$ [M+H]$^+$: 215.1231; found 215.1236.



13

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 2-iodotoluene (65.4 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **13** (27.2 mg, 65%).

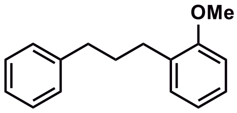
Physical state: colorless oil;

TLC: R_f = 0.56 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.25 (m, 2H), 7.22 – 7.16 (m, 3H), 7.14 – 7.08 (m, 4H), 2.74 – 2.60 (m, 4H), 2.27 (s, 3H), 1.96 – 1.86 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 142.4, 140.6, 136.0, 130.3, 128.9, 128.6, 128.5, 126.0, 125.9, 125.7, 36.0, 33.0, 31.9, 19.4;

HRMS (ESI): Calcd for $C_{16}H_{19}$ $[M+H]^+$: 211.1481; found 211.1487.



14

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 2-iodoanisole (70.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **14** (38.9 mg, 86%).

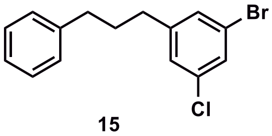
Physical state: colorless oil;

TLC: R_f = 0.63 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 – 7.25 (m, 2H), 7.22 – 7.11 (m, 5H), 6.90 – 6.82 (m, 2H), 3.81 (s, 3H), 2.70 – 2.63 (m, 4H), 1.99 – 1.86 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 157.6, 142.8, 130.9, 129.9, 128.6, 128.4, 127.1, 125.7, 120.4, 110.3, 55.4, 35.9, 31.5, 30.1;

HRMS (ESI): Calcd for $C_{16}H_{19}O$ $[M+H]^+$: 227.1430; found 227.1436.



15

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 1-bromo-3-chloro-5-iodobenzene (95.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **15** (28.5 mg, 46%).

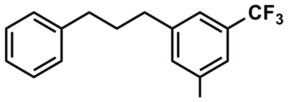
Physical state: colorless oil;

TLC: R_f = 0.55 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.34 (t, *J* = 1.9 Hz, 1H), 7.33 – 7.27 (m, 2H), 7.24 – 7.16 (m, 4H), 7.10 (t, *J* = 1.8 Hz, 1H), 2.65 (t, *J* = 7.7 Hz, 2H), 2.59 (t, *J* = 7.7 Hz, 2H), 1.98 – 1.87 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): *δ* 146.0, 141.7, 135.0, 130.0, 128.9, 128.6, 128.5, 127.6, 126.1, 122.7, 35.4, 34.9, 32.6;

HRMS (ESI): Calcd for $C_{15}H_{15}BrCl$ $[M+H]^+$: 309.0044; found 309.0046.



16

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 3-chloro-5-(trifluoromethyl) benzene iodine (91.9 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **16** (32.3 mg, 54%).

Physical state: colorless oil;

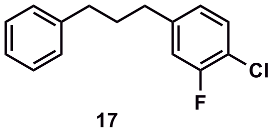
TLC: R_f = 0.83 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.44 (d, *J* = 2.0 Hz, 1H), 7.34 (d, *J* = 2.0 Hz, 1H), 7.32 – 7.29 (m, 2H), 7.28 (d, *J* = 1.5 Hz, 1H), 7.23 – 7.16 (m, 3H), 2.71 – 2.64 (m, 4H), 1.97 (tt, *J* = 9.3, 6.8 Hz, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): *δ* 145.4, 141.6, 134.8, 132.2 (q, *J* = 26.4 Hz), 132.1, 128.6, 128.5, 126.2, 123.6 (q, *J* = 3.0 Hz), 123.2 (q, *J* = 3.0 Hz), 35.4, 35.1, 32.6;

^{19}F NMR (376 MHz, $CDCl_3$): *δ* –62.77 (s);

HRMS (ESI): Calcd for $C_{16}H_{15}ClF_3$ $[M+H]^+$: 299.0809; found 299.0814.



17

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 4-chloro-3-fluoroiodobenzene (76.9 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **17** (29.8 mg, 60%).

Physical state: colorless oil;

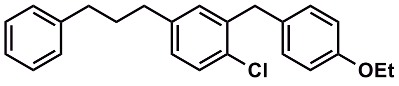
TLC: R_f = 0.75 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.32 – 7.23 (m, 3H), 7.22 – 7.14 (m, 3H), 6.96 (dt, *J* = 8.2, 1.5 Hz, 1H), 6.89 (dt, *J* = 8.2, 1.5 Hz, 1H), 2.69 – 2.54 (m, 4H), 1.98 – 1.88 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): *δ* 158.1 (d, *J* = 248.3 Hz), 143.4 (d, *J* = 6.5 Hz), 141.9, 130.4, 128.5, 126.1, 125.0 (d, *J* = 3.2 Hz), 118.2, 118.0, 116.6 (d, *J* = 20.4 Hz), 35.3, 34.8, 32.7;

^{19}F NMR (376 MHz, $CDCl_3$): *δ* –116.27 (dd, *J* = 10.2, 7.6 Hz);

HRMS (ESI): Calcd for $C_{15}H_{15}ClF$ $[M+H]^+$: 249.0841; found 249.0846.



18

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 4-iodo-1-chloro-2-(4ethoxybenzyl) benzene (111.8 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **18** (48 mg, 66%).

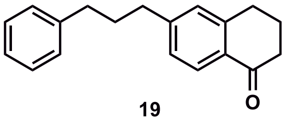
Physical state: colorless oil;

TLC: R$_f$ = 0.54 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.30 – 7.24 (m, 3H), 7.20 – 7.06 (m, 5H), 6.98 – 6.93 (m, 2H), 6.84 – 6.80 (m, 2H), 4.04 – 3.93 (m, 4H), 2.59 (t, J = 7.7 Hz, 2H), 2.55 (t, J = 7.7 Hz, 2H), 1.95 – 1.83 (m, 2H), 1.39 (t, J = 7.0 Hz, 3H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 157.5, 142.2, 141.1, 138.9, 131.7, 131.5, 131.2, 130.0, 129.4, 128.6, 128.5, 127.7, 125.9, 114.5, 63.5, 38.4, 35.4, 34.8, 32.9, 15.0;

HRMS (ESI): Calcd for $C_{24}H_{26}ClO$ [M+H]$^+$: 365.1667; found 365.1672.



19

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 6-iodo-1-tetralone (81.6 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **19** (44.8 mg, 85%).

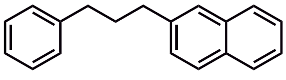
Physical state: colorless oil;

TLC: R$_f$ = 0.20 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (500 MHz, CDCl$_3$): δ 7.96 (d, J = 8.0 Hz, 1H), 7.33 – 7.27 (m, 2H), 7.23 – 7.16 (m, 3H), 7.13 (dd, J = 8.1, 1.7 Hz, 1H), 7.06 (d, J = 1.7 Hz, 1H), 2.93 (t, J = 6.1 Hz, 2H), 2.70 – 2.59 (m, 6H), 2.17 – 2.09 (m, 2H), 2.01 – 1.93 (m, 2H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 198.3, 148.6, 144.7, 142.0, 130.8, 128.7, 128.5, 128.5, 127.5, 127.1, 126.0, 39.3, 35.6, 35.5, 32.7, 29.9, 23.5;

HRMS (ESI): Calcd for $C_{19}H_{21}O$ [M+H]$^+$: 265.1587; found 265.1592.



20

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 2-iodonaphthalene (76.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **20** (34.5 mg, 70%).

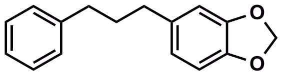
Physical state: colorless oil;

TLC: R$_f$ = 0.63 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.82 – 7.75 (m, 3H), 7.63 – 7.61 (m, 1H), 7.47 – 7.39 (m, 2H), 7.35 – 7.27 (m, 3H), 7.23 – 7.16 (m, 3H), 2.85 – 2.78 (m, 2H), 2.72 – 2.66 (m, 2H), 2.11 – 2.01 (m, 2H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 142.4, 139.9, 133.8, 132.1, 128.6, 128.5, 128.0, 127.7, 127.5, 127.5, 126.6, 126.0, 125.9, 125.2, 35.7, 35.6, 33.0;

HRMS (ESI): Calcd for $C_{19}H_{19}$ [M+H]$^+$: 247.1481; found 247.1487.



21

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 1-iodo-3,4-methylenedioxybenzene (74.4 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **21** (32.9 mg, 69%).

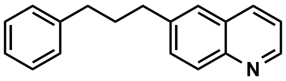
Physical state: colorless oil;

TLC: R$_f$ = 0.59 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.33 – 7.25 (m, 2H), 7.22 – 7.15 (m, 3H), 6.76 – 6.62 (m, 3H), 5.93 (d, *J* = 0.9 Hz, 2H), 2.64 (t, *J* = 7.7 Hz, 2H), 2.58 (t, *J* = 7.7 Hz, 2H), 1.98 – 1.87 (m, 2H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 147.6, 145.7, 142.4, 136.3, 128.6, 128.4, 125.9, 121.3, 109.0, 108.2, 100.9, 35.4, 35.3, 33.4;

HRMS (ESI): Calcd for $C_{16}H_{17}O_2$ [M+H]$^+$: 241.1223; found 241.1228.



22

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 6-iodoquinoline (76.5 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 4:1, Petroleum ether: EtOAc) afforded **22** (41.9 mg, 85%).

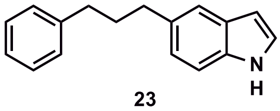
Physical state: colorless oil;

TLC: R$_f$ = 0.25 (silica gel, 4:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 8.87 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.11 – 8.02 (m, 2H), 7.60 – 7.56 (m, 2H), 7.37 (dd, *J* = 8.3, 4.3 Hz, 1H), 7.33 – 7.28 (m, 2H), 7.24 – 7.18 (m, 3H), 2.84 (t, *J* = 7.7 Hz, 2H), 2.70 (t, *J* = 7.7 Hz, 2H), 2.12 – 2.01 (m, 2H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 149.8, 147.2, 142.1, 140.8, 135.7, 131.1, 129.4, 128.6, 128.5, 128.4, 126.3, 126.0, 121.2, 35.5, 35.4, 32.8;

HRMS (ESI): Calcd for $C_{18}H_{18}N$ [M+H]$^+$: 248.1434; found 248.1439.



23

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 5-iodoindole (72.9 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 9:1, Petroleum ether: EtOAc) afforded **23** (33.7 mg, 72%).

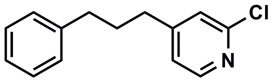
Physical state: colorless oil;

TLC: R_f = 0.20 (silica gel, 9:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (s, 1H), 7.46 – 7.44 (m, 1H), 7.33 – 7.24 (m, 3H), 7.21 – 7.15 (m, 4H), 7.04 (dd, J = 8.3, 1.7 Hz, 1H), 6.51 – 6.47 (m, 1H), 2.75 (t, J = 7.8 Hz, 2H), 2.67 (t, J = 7.8 Hz, 2H), 2.06 – 1.96 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 142.8, 134.5, 133.8, 128.6, 128.4, 128.2, 125.8, 124.4, 123.2, 120.0, 110.9, 102.4, 35.7, 35.6, 33.9;

HRMS (ESI): Calcd for $C_{17}H_{18}N$ $[M+H]^+$: 236.1434; found 236.1439.



24

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 2-chloro-4-iodopyridine (71.8 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 9:1, Petroleum ether: EtOAc) afforded **24** (27.7 mg, 60%).

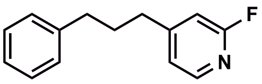
Physical state: colorless oil;

TLC: R_f = 0.24 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.29 (d, J = 5.1 Hz, 1H), 7.34 – 7.27 (m, 2H), 7.24 – 7.20 (m, 1H), 7.20 – 7.14 (m, 3H), 7.04 (dd, J = 5.1, 1.5 Hz, 1H), 2.66 (t, J = 7.8 Hz, 2H), 2.63 (t, J = 7.8 Hz, 2H), 2.01 – 1.92 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 154.9, 151.6, 149.6, 141.3, 128.6, 128.5, 126.2, 124.3, 122.8, 35.3, 34.4, 31.6;

HRMS (ESI): Calcd for $C_{14}H_{15}ClN$ $[M+H]^+$: 232.0888; found 232.0893.



25

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 2-fluoro-4-iodopyridine (66.9 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 4:1, Petroleum ether: EtOAc) afforded **25** (25.9 mg, 60%).

Physical state: colorless oil;

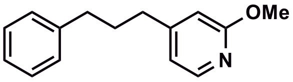
TLC: R$_f$ = 0.38 (silica gel, 4:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 8.10 (dd, J = 5.2, 0.7 Hz, 1H), 7.34 – 7.28 (m, 2H), 7.24 – 7.16 (m, 3H), 7.02 – 6.97 (m, 1H), 6.76 – 6.72 (m, 1H), 2.70 – 2.64 (m, 4H), 1.98 (tt, J = 9.2, 6.9 Hz, 2H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 164.2 (d, J = 238.3 Hz), 157.4 (d, J = 8.1 Hz), 147.4 (d, J = 15.4 Hz), 141.4, 128.6, 128.5, 126.2, 121.7 (d, J = 3.9 Hz), 109.2 (d, J = 36.6 Hz), 35.3, 34.6 (d, J = 2.9 Hz), 31.7;

^{19}F NMR (376 MHz, CDCl$_3$): δ –69.17 (s);

HRMS (ESI): Calcd for $C_{14}H_{15}FN$ [M+H]$^+$: 216.1183; found 216.1188.



26

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 4-iodo-2-methoxypyridine (70.5 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **26** (30.8 mg, 68%).

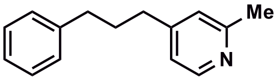
Physical state: colorless oil;

TLC: R$_f$ = 0.40 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 8.05 (dd, J = 5.3, 0.7 Hz, 1H), 7.32 – 7.23 (m, 2H), 7.22 – 7.14 (m, 3H), 6.71 (dd, J = 5.3, 1.4 Hz, 1H), 6.57 (dd, J = 1.5, 0.8 Hz, 1H), 3.93 (s, 3H), 2.64 (t, J = 7.7 Hz, 2H), 2.59 (t, J = 7.7 Hz, 2H), 1.99 – 1.89 (m, 2H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 164.6, 154.2, 146.6, 141.9, 128.6, 128.5, 126.1, 117.7, 110.4, 53.5, 35.4, 34.7, 31.8;

HRMS (ESI): Calcd for $C_{15}H_{18}NO$ [M+H]$^+$: 228.1383; found 228.1388.



27

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 4-iodo-2-methylpyridine (65.7 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **27** (26 mg, 62%).

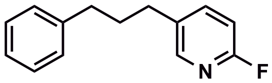
Physical state: colorless oil;

TLC: R$_f$ = 0.41 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 8.39 (dd, J = 5.1, 0.8 Hz, 1H), 7.33 – 7.26 (m, 2H), 7.25 – 7.17 (m, 3H), 6.99 – 6.97 (m, 1H), 6.95 – 6.91 (m, 1H), 2.66 (t, J = 7.7 Hz, 2H), 2.61 (t, J = 7.7 Hz, 2H), 2.53 (s, 3H), 2.01 – 1.92 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): *δ* 158.3, 151.6, 149.0, 141.8, 128.5, 126.1, 123.5, 121.1, 35.4, 34.7, 31.9, 24.4;

HRMS (ESI): Calcd for $C_{15}H_{18}N$ $[M+H]^+$: 212.1434; found 212.1439.



28

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 2-fluoro-5-iodopyridine (66.9 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether) afforded **28** (14.7 mg, 34%).

Physical state: colorless oil;

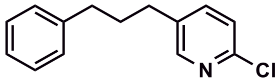
TLC: R_f = 0.38 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): *δ* 8.03 – 7.99 (m, 1H), 7.58 (td, *J* = 8.1, 2.6 Hz, 1H), 7.32 – 7.26 (m, 2H), 7.22 – 7.14 (m, 3H), 6.84 (dd, *J* = 8.4, 3.0 Hz, 1H), 2.68 – 2.59 (m, 4H), 1.99 – 1.89 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): *δ* 162.4 (d, *J* = 237.2 Hz), 147.1 (d, *J* = 14.1 Hz), 141.6, 141.1 (d, *J* = 7.6 Hz), 135.1 (d, *J* = 4.7 Hz), 128.6, 128.5, 126.1, 109.2 (d, *J* = 37.1 Hz), 35.3, 32.8, 31.5;

^{19}F NMR (376 MHz, $CDCl_3$): *δ* –72.27 (d, *J* = 5.4 Hz);

HRMS (ESI): Calcd for $C_{14}H_{15}FN$ $[M+H]^+$: 216.1183; found 216.1188.



29

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 2-chloro-5-iodopyridine (71.8 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **29** (25.3 mg, 55%).

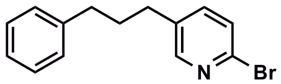
Physical state: colorless oil;

TLC: R_f = 0.35 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): *δ* 8.20 (dd, *J* = 2.5, 0.7 Hz, 1H), 7.45 (dd, *J* = 8.1, 2.5 Hz, 1H), 7.31 – 7.26 (m, 2H), 7.25 – 7.14 (m, 4H), 2.65 (t, *J* = 7.7 Hz, 2H), 2.61 (t, *J* = 7.7 Hz, 2H), 1.98 – 1.89 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): *δ* 149.7, 149.1, 141.6, 138.9, 136.5, 128.6, 128.3, 126.2, 124.0, 35.3, 32.6, 31.7;

HRMS (ESI): Calcd for $C_{14}H_{15}ClN$ $[M+H]^+$: 232.0888; found 232.0893.



30

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 2-bromo-5-iodopyridine (85.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **30** (19 mg, 35%).

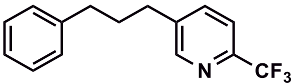
Physical state: colorless oil;

TLC: R_f = 0.24 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.21 – 8.18 (m, 1H), 7.42 – 7.33 (m, 2H), 7.33 – 7.25 (m, 2H), 7.23 – 7.15 (m, 3H), 2.65 (t, *J* = 7.7 Hz, 2H), 2.60 (t, *J* = 7.7 Hz, 2H), 1.95 (tt, *J* = 9.1, 6.9 Hz, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 150.3, 141.5, 139.5, 138.7, 136.9, 128.6, 128.5, 127.8, 126.2, 35.3, 32.5, 31.8;

HRMS (ESI): Calcd for $C_{14}H_{15}BrN$ $[M+H]^+$: 276.0382; found 276.0388.



31

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 5-iodo-2-(trifluoromethyl) pyridine (81.9 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **31** (27.2 mg, 51%).

Physical state: colorless oil;

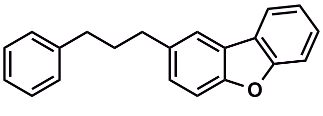
TLC: R_f = 0.39 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55 (d, *J* = 2.1 Hz, 1H), 7.68 – 7.63 (m, 1H), 7.62 – 7.57 (m, 1H), 7.33 – 7.27 (m, 2H), 7.24 – 7.15 (m, 3H), 2.72 (t, *J* = 7.7 Hz, 2H), 2.68 (t, *J* = 7.7 Hz, 2H), 1.99 (tt, *J* = 9.0, 6.9 Hz, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 150.3, 146.1 (q, *J* = 34.8 Hz), 141.4, 141.0, 137.1, 128.6, 128.5, 126.2, 121.8 (q, *J* = 273.7 Hz), 120.3 (q, *J* = 3.0 Hz), 35.3, 32.4, 32.4;

^{19}F NMR (376 MHz, $CDCl_3$): δ –67.69 (s);

HRMS (ESI): Calcd for $C_{15}H_{15}F_3N$ $[M+H]^+$: 266.1151; found 266.1157.



32

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 2-iododibenzofuran (88.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **32** (43.8 mg, 77%).

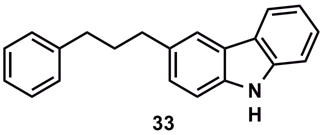
Physical state: colorless oil;

TLC: R_f = 0.83 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (ddd, *J* = 7.7, 1.4, 0.7 Hz, 1H), 7.75 (d, *J* = 1.7 Hz, 1H), 7.58 – 7.54 (m, 1H), 7.50 – 7.40 (m, 2H), 7.36 – 7.25 (m, 4H), 7.24 – 7.19 (m, 3H), 2.81 (t, *J* = 7.8 Hz, 2H), 2.70 (t, *J* = 7.8 Hz, 2H), 2.11 – 2.01 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 156.6, 154.8, 142.4, 136.9, 128.6, 128.5, 127.8, 127.1, 125.9, 124.4, 124.3, 122.7, 120.7, 120.2, 111.8, 111.4, 35.5, 35.5, 33.7;

HRMS (ESI): Calcd for $C_{21}H_{19}O$ $[M+H]^+$: 287.1430; found 287.1436.



33

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 3-iodo-9H-carbazole (87.9 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 4:1, Petroleum ether: EtOAc) afforded **33** (42.5 mg, 75%).

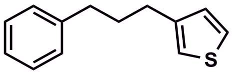
Physical state: colorless oil;

TLC: R_f = 0.60 (silica gel, 4:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.05 (d, *J* = 7.8 Hz, 1H), 7.97 (s, 1H), 7.89 (d, *J* = 1.6 Hz, 1H), 7.44 – 7.39 (m, 2H), 7.35 (d, *J* = 8.2 Hz, 1H), 7.32 – 7.26 (m, 3H), 7.25 – 7.18 (m, 4H), 2.84 (t, *J* = 7.7 Hz, 2H), 2.71 (t, *J* = 7.7 Hz, 2H), 2.11 – 2.01 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 142.6, 139.9, 138.0, 133.5, 128.6, 128.4, 126.7, 125.8, 125.8, 123.5, 123.3, 120.3, 119.8, 119.3, 110.7, 110.5, 35.6, 35.6, 33.9;

HRMS (ESI): Calcd for $C_{21}H_{20}N$ $[M+H]^+$: 286.1590; found 286.1596.



34

On 0.2 mmol scale, **General Procedure** was followed with phenylbutyric acid (32.8 mg, 0.2 mmol, 1.0 equiv), 3-iodothiophene (63.0 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **34** (16.7 mg, 41%).

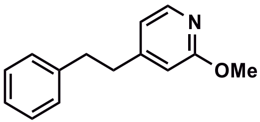
Physical state: colorless oil;

TLC: R_f = 0.73 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 – 7.23 (m, 3H), 7.21 – 7.15 (m, 3H), 6.95 – 6.93 (m, 2H), 2.70 – 2.62 (m, 4H), 2.02 – 1.91 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 142.7, 142.3, 128.6, 128.5, 128.3, 125.9, 125.4, 120.2, 35.6, 32.2, 29.9;

HRMS (ESI): Calcd for $C_{13}H_{15}S$ $[M+H]^+$: 203.0889; found 203.0894.



35

On 0.2 mmol scale, **General Procedure** was followed with 3-phenylpropionic acid (30.0 mg, 0.2 mmol, 1.0 equiv), 4-iodo-2-methoxypyridine (70.5 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **35** (29 mg, 68%).

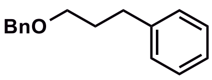
Physical state: colorless oil;

TLC: R_f = 0.30 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 (dd, J = 5.3, 0.8 Hz, 1H), 7.32 – 7.26 (m, 2H), 7.24 – 7.14 (m, 3H), 6.72 – 6.69 (m, 1H), 6.58 – 6.55 (m, 1H), 3.93 (s, 3H), 2.96 – 2.84 (m, 4H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.6, 153.6, 146.7, 141.0, 128.6, 128.5, 126.3, 117.7, 110.4, 53.5, 37.1, 36.5;

HRMS (ESI): Calcd for $C_{14}H_{16}NO$ $[M+H]^+$: 214.1226; found 214.1232.



36

On 0.2 mmol scale, **General Procedure** was followed with 4-benzyloxybutyric acid (38.8 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **36** (32.6 mg, 72%).

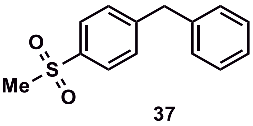
Physical state: colorless oil;

TLC: R_f = 0.52 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 – 7.34 (m, 4H), 7.32 – 7.24 (m, 3H), 7.20 – 7.16 (m, 3H), 4.51 (s, 2H), 3.50 (td, J = 6.4, 0.7 Hz, 2H), 2.72 (dd, J = 8.7, 6.8 Hz, 2H), 1.99 – 1.89 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 142.1, 138.7, 128.6, 128.5, 128.5, 127.8, 127.7, 125.9, 73.1, 69.6, 32.5, 31.5;

HRMS (ESI): Calcd for $C_{16}H_{19}O$ $[M+H]^+$: 227.1430; found 227.1436.



37

On 0.2 mmol scale, **General Procedure** was followed with 4-methylsulphonyl-phenylacetic acid (42.8 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 3:2, Petroleum ether: EtOAc) afforded **37** (30 mg, 61%).

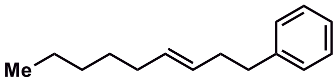
Physical state: colorless oil;

TLC: R_f = 0.60 (silica gel, 3:2, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.87 – 7.84 (m, 2H), 7.78 – 7.75 (m, 1H), 7.40 – 7.37 (m, 2H), 7.34 – 7.29 (m, 2H), 7.20 – 7.15 (m, 2H), 4.07 (s, 2H), 3.03 (s, 3H);

^{13}C NMR (126 MHz, CDCl$_3$): δ 147.8, 139.5, 138.4, 129.9, 129.1, 128.9, 127.7, 126.8, 44.7, 41.9;

HRMS (ESI): Calcd for $C_{14}H_{15}O_2S$ $[M+H]^+$: 247.0787; found 247.0793.



38

On 0.2 mmol scale, **General Procedure** was followed with trans-4-decenoic acid (34.1 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **38** (25 mg, 62%).

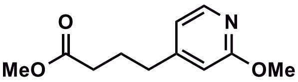
Physical state: colorless oil;

TLC: $R_f = 0.90$ (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.33 – 7.23 (m, 2H), 7.23 – 7.13 (m, 3H), 5.49 – 5.37 (m, 2H), 2.67 (dd, J = 9.1, 6.6 Hz, 2H), 2.35 – 2.25 (m, 2H), 2.02 – 1.91 (m, 2H), 1.37 – 1.23 (m, 6H), 0.89 (t, J = 7.0 Hz, 3H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 142.4, 131.4, 129.4, 128.6, 128.4, 125.8, 36.3, 34.6, 32.7, 31.5, 29.4, 22.7, 14.2;

HRMS (ESI): Calcd for $C_{15}H_{23}$ $[M+H]^+$: 203.1794; found 203.1800.



39

On 0.2 mmol scale, **General Procedure** was followed with mono-methyl glutarate (29.2 mg, 0.2 mmol, 1.0 equiv), 4-iodo-2-methoxypyridine (70.5 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **39** (17.6 mg, 42%).

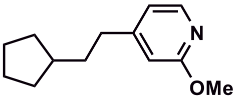
Physical state: colorless oil;

TLC: $R_f = 0.15$ (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (500 MHz, CDCl$_3$): δ 8.06 (d, J = 5.3 Hz, 1H), 6.71 (dd, J = 5.2, 1.4 Hz, 1H), 6.58 – 6.54 (m, 1H), 3.92 (s, 3H), 3.67 (s, 3H), 2.60 (t, J = 7.2 Hz, 2H), 2.33 (t, J = 7.4 Hz, 2H), 2.00 – 1.90 (m, 2H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 173.7, 164.6, 153.3, 146.7, 117.6, 110.5, 53.5, 51.7, 34.4, 33.3, 25.3;

HRMS (ESI): Calcd for $C_{11}H_{16}NO_3$ $[M+H]^+$: 210.1125; found 210.1130.



40

On 0.2 mmol scale, **General Procedure** was followed with 3-cyclopentylpropionic acid (28.4 mg, 0.2 mmol, 1.0 equiv), 4-iodo-2-methoxypyridine (70.5 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **40** (21.5 mg, 52%).

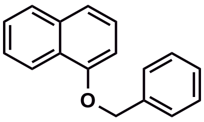
Physical state: colorless oil;

TLC: R_f = 0.40 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, J = 5.3 Hz, 1H), 6.71 (dd, J = 5.3, 1.4 Hz, 1H), 6.56 (dd, J = 1.5, 0.8 Hz, 1H), 3.92 (s, 3H), 2.59 – 2.52 (m, 2H), 1.83 – 1.71 (m, 3H), 1.65 – 1.56 (m, 4H), 1.55 – 1.47 (m, 2H), 1.17 – 1.06 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.5, 155.1, 146.4, 117.7, 110.3, 53.5, 39.6, 36.8, 34.5, 32.7, 25.3;

HRMS (ESI): Calcd for $C_{13}H_{20}NO$ $[M+H]^+$: 206.1539; found 206.1545.



41

On 0.2 mmol scale, **General Procedure** was followed with 1-naphthoxyacetic acid (40.4 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **41** (30.1 mg, 64%).

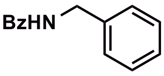
Physical state: colorless oil;

TLC: R_f = 0.71 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.39 – 8.36 (m, 1H), 7.84 – 7.80 (m, 1H), 7.57 – 7.53 (m, 2H), 7.52 – 7.48 (m, 2H), 7.48 – 7.41 (m, 3H), 7.41 – 7.35 (m, 2H), 6.90 (dd, J = 7.6, 1.0 Hz, 1H), 5.27 (s, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 154.6, 137.3, 134.7, 128.7, 128.1, 127.6, 127.5, 126.6, 126.0, 125.9, 125.4, 122.3, 120.6, 105.3, 70.2;

HRMS (ESI): Calcd for $C_{17}H_{15}O$ $[M+H]^+$: 235.1117; found 235.1123.



42

On 0.2 mmol scale, **General Procedure** was followed with hippuric acid (35.8 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 4:1, Petroleum ether: EtOAc) afforded **42** (34.2 mg, 81%).

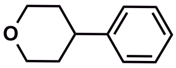
Physical state: colorless oil;

TLC: $R_f = 0.25$ (silica gel, 4:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 – 7.80 (m, 2H), 7.54 – 7.49 (m, 1H), 7.47 – 7.42 (m, 2H), 7.40 – 7.36 (m, 4H), 7.35 – 7.29 (m, 1H), 6.49 (s, 1H), 4.67 (d, *J* = 5.7 Hz, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.5, 138.3, 134.5, 131.7, 128.9, 128.7, 128.1, 127.8, 127.1, 44.3;

HRMS (ESI): Calcd for $C_{14}H_{14}NO$ $[M+H]^+$: 212.1070; found 212.1075.



43

On 0.2 mmol scale, **General Procedure** was followed with tetrahydro-2H-pyran-4-carboxylic acid (26.0 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **43** (21.6 mg, 67%).

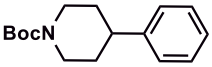
Physical state: colorless oil;

TLC: $R_f = 0.42$ (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 – 7.28 (m, 2H), 7.24 – 7.18 (m, 3H), 4.11 – 4.02 (m, 2H), 3.56 – 3.47 (m, 2H), 2.79 – 2.69 (m, 1H), 1.89 – 1.71 (m, 4H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 146.0, 128.7, 126.9, 126.5, 68.6, 41.7, 34.1;

HRMS (ESI): Calcd for $C_{11}H_{15}O$ $[M+H]^+$: 163.1117; found 163.1123.



44

On 0.2 mmol scale, **General Procedure** was followed with *N*-Boc-piperidine-4-carboxylic acid (45.9 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 9:1, Petroleum ether: EtOAc) afforded **44** (32.6 mg, 62%).

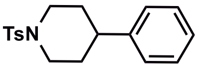
Physical state: yellow oil;

TLC: $R_f = 0.30$ (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 – 7.27 (m, 2H), 7.22 – 7.17 (m, 3H), 4.28 – 4.20 (m, 2H), 2.87 – 2.74 (m, 2H), 2.68 – 2.58 (m, 1H), 1.86 – 1.77 (m, 2H), 1.68 – 1.56 (m, 2H), 1.48 (s, 9H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 155.0, 145.9, 128.6, 126.9, 126.4, 79.5, 44.5, 42.8, 33.3, 28.6;

HRMS (ESI): Calcd for $C_{16}H_{24}NO_2$ $[M+H]^+$: 262.1802; found 262.1807.



45

On 0.2 mmol scale, **General Procedure** was followed with 1-[(4-methylphenyl)sulfonyl]-4-piperidine carboxylic acid (56.7 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 4:1, Petroleum ether: EtOAc) afforded **45** (53 mg, 83%).

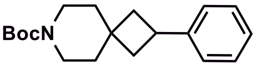
Physical state: colorless oil;

TLC: R_f = 0.50 (silica gel, 4:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 – 7.67 (m, 2H), 7.38 – 7.27 (m, 4H), 7.24 – 7.18 (m, 1H), 7.17 – 7.12 (m, 2H), 4.05 – 3.84 (m, 2H), 2.45 (s, 3H), 2.44 – 2.30 (m, 3H), 1.91 – 1.81 (m, 4H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 145.0, 143.6, 133.2, 129.8, 128.7, 127.9, 126.8, 126.7, 47.0, 41.9, 32.6, 21.7;

HRMS (ESI): Calcd for $C_{18}H_{22}NO_2S$ $[M+H]^+$: 316.1366; found 316.1371.



46

On 0.2 mmol scale, **General Procedure** was followed with 7-(tert-butoxycarbonyl)-7-azaspiro[3.5]nonane-2-carboxylic acid (53.9 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 9:1, Petroleum ether: EtOAc) afforded **46** (39.8 mg, 66%).

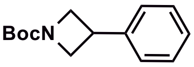
Physical state: colorless oil;

TLC: R_f = 0.30 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 – 7.27 (m, 2H), 7.23 – 7.15 (m, 3H), 3.52 (t, J = 9.1 Hz, 1H), 3.45 – 3.40 (m, 2H), 3.33 – 3.27 (m, 2H), 2.33 – 2.25 (m, 2H), 1.90 (td, J = 9.5, 2.5 Hz, 2H), 1.73 – 1.68 (m, 2H), 1.53 – 1.44 (m, 2H), 1.46 (s, 9H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 155.1, 146.1, 128.4, 126.5, 125.9, 79.3, 41.1, 40.7, 39.3, 39.2, 36.0, 33.9, 33.5, 28.6;

HRMS (ESI): Calcd for $C_{19}H_{28}NO_2$ $[M+H]^+$: 302.2115; found 302.2120.



47

On 0.2 mmol scale, **General Procedure** was followed with 1-N-Boc-3-azetidine-carboxylic acid (40.2 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 9:1, Petroleum ether: EtOAc) afforded **47** (24.3 mg, 52%).

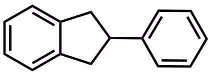
Physical state: colorless oil;

TLC: R$_f$ = 0.30 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.37 – 7.28 (m, 4H), 7.27 – 7.22 (m, 1H), 4.32 (t, J = 8.9 Hz, 2H), 3.97 (dd, J = 8.8, 6.0 Hz, 2H), 3.72 (tt, J = 8.8, 6.0 Hz, 1H), 1.46 (s, 9H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 156.5, 142.4, 128.8, 127.1, 126.9, 79.6, 56.7, 33.6, 28.5;

HRMS (ESI): Calcd for $C_{14}H_{20}NO_2$ [M+H]$^+$: 234.1489; found 234.1494.



48

On 0.2 mmol scale, **General Procedure** was followed with 2-indanancarboxylic acid (32.4 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **48** (27.6 mg, 71%).

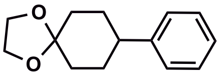
Physical state: colorless oil;

TLC: R$_f$ = 0.81 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.32 (d, J = 4.4 Hz, 4H), 7.28 – 7.23 (m, 3H), 7.23 – 7.18 (m, 2H), 3.76 – 3.66 (m, 1H), 3.40 – 3.32 (m, 2H), 3.10 (dd, J = 15.5, 8.9 Hz, 2H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 145.6, 143.1, 128.6, 127.2, 126.6, 126.3, 124.5, 45.6, 41.4;

HRMS (ESI): Calcd for $C_{15}H_{15}$ [M+H]$^+$: 195.1168; found 195.1174.



49

On 0.2 mmol scale, **General Procedure** was followed with 1,4-dioxaspiro[4.5]decane-8-carboxylic acid (37.2 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **49** (27.2 mg, 62%).

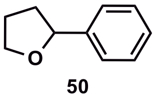
Physical state: colorless oil;

TLC: R$_f$ = 0.30 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.32 – 7.27 (m, 2H), 7.26 – 7.23 (m, 2H), 7.22 – 7.17 (m, 1H), 3.99 (s, 4H), 2.57 (tt, J = 11.6, 3.6 Hz, 1H), 1.93 – 1.65 (m, 8H);

^{13}C NMR (101 MHz, CDCl$_3$): δ 146.7, 128.5, 127.0, 126.2, 108.6, 64.5, 64.4, 43.5, 35.3, 31.7;

HRMS (ESI): Calcd for $C_{14}H_{19}O_2$ [M+H]$^+$: 219.1380; found 219.1385.



50

On 0.2 mmol scale, **General Procedure** was followed with 2-tetrahydrofuroic acid (23.2 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **50** (18.5 mg, 63%).

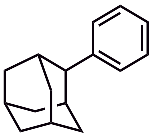
Physical state: colorless oil;

TLC: R_f = 0.51 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 – 7.32 (m, 4H), 7.29 – 7.20 (m, 1H), 4.89 (t, J = 7.2 Hz, 1H), 4.10 (dt, J = 8.1, 6.8 Hz, 1H), 3.94 (ddd, J = 8.2, 7.5, 6.4 Hz, 1H), 2.37 – 2.27 (m, 1H), 2.06 – 1.94 (m, 2H), 1.85 – 1.76 (m, 1H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 143.6, 128.4, 127.2, 125.8, 80.8, 68.8, 34.7, 26.2;

HRMS (ESI): Calcd for $C_{10}H_{13}O$ $[M+H]^+$: 149.0961; found 149.0966.



51

On 0.2 mmol scale, **General Procedure** was followed with 2-adamantanecarboxylic acid (36.0 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **51** (14.9 mg, 35%).

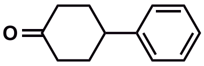
Physical state: colorless oil;

TLC: R_f = 0.83 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 – 7.28 (m, 4H), 7.20 – 7.14 (m, 1H), 3.00 (s, 1H), 2.49 – 2.45 (m, 2H), 2.04 – 1.90 (m, 5H), 1.87 – 1.75 (m, 5H), 1.58 – 1.52 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 144.6, 128.3, 127.0, 125.3, 46.9, 39.3, 38.1, 32.1, 31.2, 28.2, 27.9;

HRMS (ESI): Calcd for $C_{16}H_{21}$ $[M+H]^+$: 213.1638; found 213.1643.



52

On 0.2 mmol scale, **General Procedure** was followed with 4-oxocyclohexanecarboxylic acid (28.4 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **52** (18 mg, 52%).

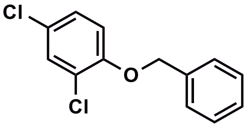
Physical state: colorless oil;

TLC: R_f = 0.20 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 – 7.29 (m, 2H), 7.28 – 7.19 (m, 3H), 3.03 (tt, J = 12.1, 3.5 Hz, 1H), 2.56 – 2.48 (m, 4H), 2.28 – 2.18 (m, 2H), 2.04 – 1.88 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 211.3, 144.9, 128.7, 126.8, 126.7, 42.9, 41.5, 34.1;

HRMS (ESI): Calcd for $C_{12}H_{15}O$ $[M+H]^+$: 175.1117; found 175.1123.



53

On 0.2 mmol scale, **General Procedure** was followed with 2,4-dichlorophenoxyacetic acid (44.2 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **53** (41 mg, 81%).

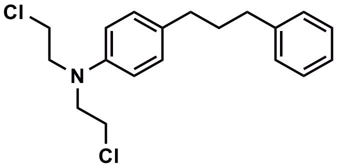
Physical state: colorless oil;

TLC: R_f = 0.60 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 – 7.31 (m, 6H), 7.15 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.88 (d, *J* = 8.8 Hz, 1H), 5.14 (s, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 153.1, 136.2, 130.2, 128.8, 128.3, 127.6, 127.2, 126.2, 124.2, 114.9, 71.2;

HRMS (ESI): Calcd for $C_{13}H_{11}Cl_2O$ [M+H]$^+$: 253.0181; found 253.0187.



54

On 0.2 mmol scale, **General Procedure** was followed with chlorambucil (60.8 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **54** (39.2 mg, 59%).

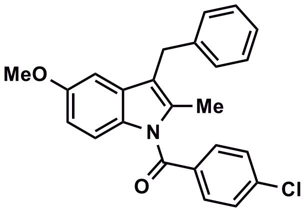
Physical state: colorless oil;

TLC: R_f = 0.40 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 – 7.25 (m, 2H), 7.21 – 7.17 (m, 3H), 7.10 – 7.06 (m, 2H), 6.65 – 6.61 (m, 2H), 3.74 – 3.58 (m, 8H), 2.64 (t, *J* = 7.7 Hz, 2H), 2.56 (t, *J* = 7.7 Hz, 2H), 1.96 – 1.87 (m, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 144.2, 142.5, 131.9, 129.8, 128.6, 128.4, 125.8, 112.4, 53.8, 40.6, 35.5, 34.4, 33.3;

HRMS (ESI): Calcd for $C_{19}H_{24}Cl_2N$ [M+H]$^+$: 336.1280; found 336.1286.



55

On 0.2 mmol scale, **General Procedure** was followed with indomethacin (71.6 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **55** (36 mg, 46%).

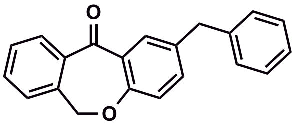
Physical state: colorless oil;

TLC: R_f = 0.36 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 – 7.64 (m, 2H), 7.50 – 7.43 (m, 2H), 7.30 – 7.16 (m, 5H), 6.89 (dd, *J* = 9.0, 0.5 Hz, 1H), 6.81 (d, *J* = 2.5 Hz, 1H), 6.64 (dd, *J* = 9.0, 2.6 Hz, 1H), 4.04 (s, 2H), 3.74 (s, 3H), 2.39 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 168.5, 156.0, 139.9, 139.2, 135.1, 134.3, 131.4, 131.2, 131.2, 129.2, 128.6, 128.3, 126.3, 118.4, 115.0, 111.2, 102.0, 55.7, 30.1, 13.6;

HRMS (ESI): Calcd for $C_{24}H_{21}ClNO_2$ $[M+H]^+$: 390.1255; found 390.1261.



56

On 0.2 mmol scale, **General Procedure** was followed with isoxepac (53.7 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **56** (31.1 mg, 52%).

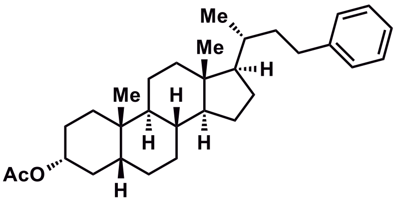
Physical state: colorless oil;

TLC: R_f = 0.25 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (d, *J* = 2.4 Hz, 1H), 7.90 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.55 (td, *J* = 7.5, 1.4 Hz, 1H), 7.47 (td, *J* = 7.5, 1.4 Hz, 1H), 7.36 (dd, *J* = 7.3, 1.3 Hz, 1H), 7.32 – 7.27 (m, 3H), 7.23 – 7.19 (m, 3H), 6.98 (d, *J* = 8.4 Hz, 1H), 5.17 (s, 2H), 3.99 (s, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 191.3, 160.0, 140.9, 140.7, 136.3, 135.8, 135.1, 132.8, 131.8, 129.6, 129.3, 129.0, 128.7, 127.9, 126.4, 125.2, 121.0, 73.7, 41.1;

HRMS (ESI): Calcd for $C_{21}H_{17}O_2$ $[M+H]^+$: 301.1223; found 301.1228.



57

On 0.2 mmol scale, **General Procedure** was followed with acetyllithocholic acid (83.7 mg, 0.2 mmol, 1.0 equiv), iodobenzene (61.2 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 19:1, Petroleum ether: EtOAc) afforded **57** (40 mg, 44%).

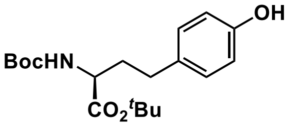
Physical state: colorless oil;

TLC: R_f = 0.45 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.30 – 7.23 (m, 2H), 7.20 – 7.12 (m, 3H), 4.77 – 4.67 (m, 1H), 2.69 (ddd, *J* = 13.5, 11.4, 4.8 Hz, 1H), 2.45 (ddd, *J* = 13.5, 11.0, 5.8 Hz, 1H), 2.02 (s, 3H), 2.01 – 1.96 (m, 5H), 1.91 – 1.63 (m, 2H), 1.62 – 1.50 (m, 2H), 1.49 – 1.33 (m, 8H), 1.31 – 1.03 (m, 9H), 1.01 (d, *J* = 6.5 Hz, 3H), 0.93 (d, *J* = 0.8 Hz, 3H), 0.64 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$): *δ* 170.8, 143.5, 128.5, 128.4, 125.6, 74.5, 56.6, 56.2, 42.9, 42.0, 40.6, 40.3, 38.3, 35.9, 35.8, 35.2, 34.7, 32.7, 32.4, 28.4, 27.2, 26.8, 26.5, 24.3, 23.5, 21.6, 21.0, 18.8, 12.2;

HRMS (ESI): Calcd for $C_{31}H_{47}O_2$ $[M+H]^+$: 451.3571; found 451.3576.



58

On 0.2 mmol scale, **General Procedure** was followed with Boc-L-glutamic acid 1-*tert*-butyl ester (60.7 mg, 0.2 mmol, 1.0 equiv), 4-iodophenol (66 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 3:1, Hexane: EtOAc) afforded **58** (34.6 mg, 49%).

Physical state: colorless oil;

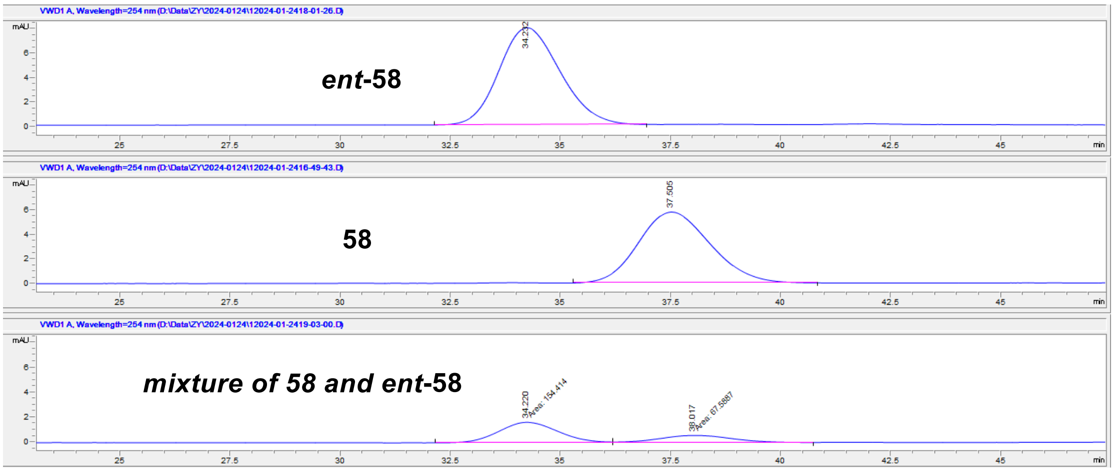
TLC: R_f = 0.60 (silica gel, 3:2, Petroleum ether: EtOAc, UV);

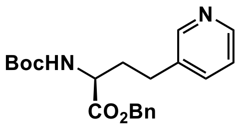
^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.00 (d, *J* = 8.5 Hz, 2H), 6.75 (d, *J* = 8.5 Hz, 2H), 5.85 (s, 1H), 5.13 (d, *J* = 8.3 Hz, 1H), 4.22 – 4.17 (m, 1H), 2.66 – 2.47 (m, 2H), 2.11 – 1.97 (m, 1H), 1.90 – 1.81 (m, 1H), 1.47 (s, 9H), 1.45 (s, 9H);

^{13}C NMR (101 MHz, $CDCl_3$): *δ* 172.1, 155.7, 154.4, 132.9, 129.5, 115.5, 82.2, 80.1, 54.0, 35.1, 30.8, 28.5, 28.1;

HRMS (ESI): Calcd for $C_{19}H_{30}NO_5$ $[M+H]^+$: 352.2118; found 352.2124.

The *ee* value of **58** was determined by HPLC on a CHIRALPAK AD-H column (10% ^{i}PrOH/hexane, 1.0 mL/min, λ = 254 nm), which show that the *ee* value of **58** was completely retained in our reaction conditions. The retention time for compound obtained from Boc-*L*-glutamic acid 1-*tert*-butyl ester: 37.5 min; The retention time for compound obtained from Boc-*D*-glutamic acid 1-tert-butyl ester: 34.2 min.





59

On 0.2 mmol scale, **General Procedure** was followed Boc-L-Glutamic acid 1-benzyl ester (67.5 mg, 0.2 mmol, 1.0 equiv), 3-iodopyridine (61.5 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, 3:1, Hexane: EtOAc) afforded **59** (41.4 mg, 56%).

Physical state: colorless oil;

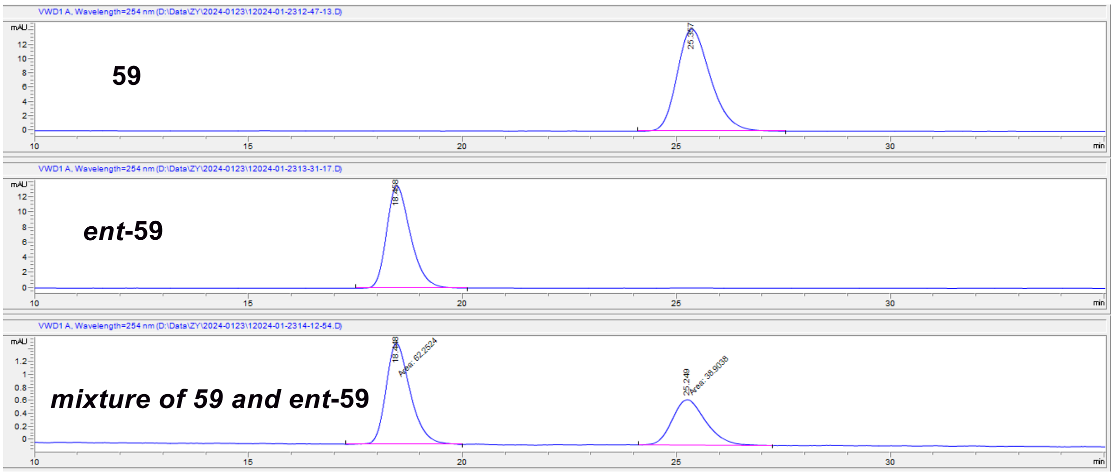
TLC: $R_f = 0.25$ (silica gel, 3:2, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.47 – 8.41 (m, 1H), 8.38 – 8.34 (m, 1H), 7.42 (dt, J = 7.8, 2.0 Hz, 1H), 7.40 – 7.30 (m, 5H), 7.18 (dd, J = 7.8, 4.8 Hz, 1H), 5.26 – 5.05 (m, 3H), 4.45 – 4.35 (m, 1H), 2.72 – 2.50 (m, 2H), 2.17 – 2.10 (m, 1H), 1.99 – 1.87 (m, 1H), 1.44 (s, 9H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 172.3, 155.4, 149.9, 147.7, 136.2, 136.0, 135.3, 128.8, 128.7, 128.6, 123.5, 80.2, 67.4, 53.2, 34.2, 28.8, 28.4;

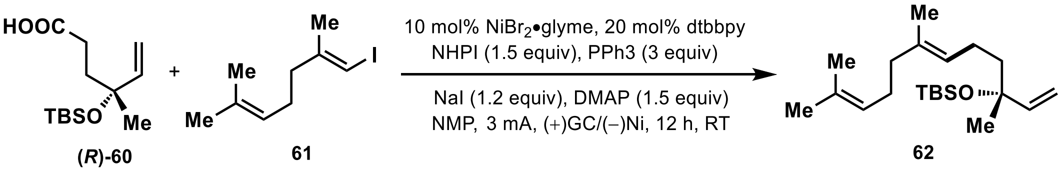
HRMS (ESI): Calcd for $C_{21}H_{27}N_2O_4$ $[M+H]^+$: 371.1965; found 371.1971.

The ee was determined via HPLC on a CHIRALPAK AD-H column (10% i-PrOH/hexane, 1.0 mL/min, λ = 254 nm), which show that the *ee* value of **59** was completely retained in our reaction conditions. The retention time for compound obtained from Boc-*L*-Glutamic acid 1-benzyl ester: 25.4 min. The retention time for compound obtained from Boc-*D*-Glutamic acid 1-benzyl ester: 18.4 min.



Decarboxylative alkenylation

The substrate carboxylic acids and vinyl iodides were prepared according to Baran's procedure with slight modification.[3-4]



On 0.2 mmol scale, **General Procedure** was followed with acid **(*R*)-60** (51.6 mg, 0.2 mmol, 1.0 equiv), vinyl iodide **61** (75 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, Petroleum ether) afforded **62** (27.7 mg, 41%).

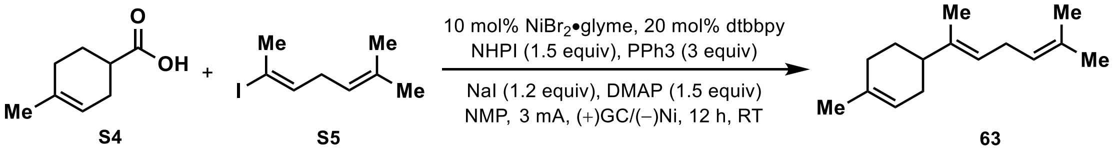
Physical state: colorless oil;

TLC: $R_f = 0.95$ (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 5.89 – 5.80 (m, 1H), 5.18 – 5.06 (m, 3H), 5.00 – 4.95 (m, 1H), 2.10 – 1.94 (m, 6H), 1.68 (s, 3H), 1.60 (s, 3H), 1.58 (s, 3H), 1.50 – 1.42 (m, 2H), 1.30 (s, 3H), 0.90 – 0.87 (m, 9H), 0.10 – 0.06 (m, 6H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 144.8, 133.9, 130.4, 123.8, 123.5, 110.7, 74.7, 42.9, 38.9, 26.6, 25.9, 25.1, 24.9, 21.8, 17.5, 16.8, 15.1, –2.9;

HRMS (ESI): Calcd for $C_{21}H_{41}OSi$ $[M+H]^+$: 337.2921; found 337.2927.



On 0.2 mmol scale, **General Procedure** was followed with acid **S8** (28 mg, 0.2 mmol, 1.0 equiv), vinyl iodide **S9** (70.8 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6

mmol, 3.0 equiv). Purification by preparative TLC (silica gel, Petroleum ether) afforded **63** (29 mg, 70%).

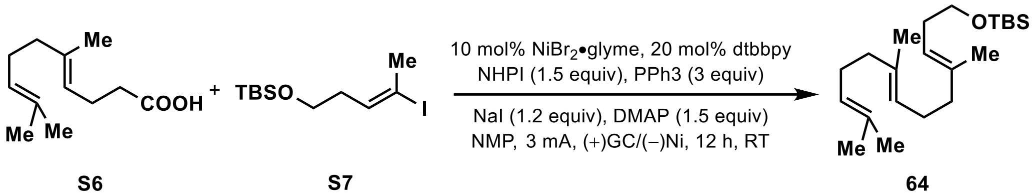
Physical state: colorless oil;

TLC: R$_f$ = 0.95 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 5.41 – 5.36 (m, 1H), 5.16 – 5.07 (m, 2H), 2.69 (t, J = 7.2 Hz, 2H), 2.11 – 1.85 (m, 5H), 1.75 – 1.70 (m, 1H), 1.69 (d, J = 1.4 Hz, 3H), 1.64 (t, J = 1.7 Hz, 3H), 1.63 (d, J = 1.2 Hz, 3H), 1.61 (d, J = 1.2 Hz, 3H), 1.52 – 1.42 (m, 1H);

^{13}C NMR (101 MHz, CDCl3): δ 139.4, 133.9, 131.4, 123.6, 121.9, 121.1, 43.0, 30.9, 30.9, 28.1, 27.1, 25.9, 23.7, 17.9, 14.3;

HRMS (ESI): Calcd for $C_{15}H_{25}$ [M+H]$^+$: 205.1951; found 205.1956.



On 0.2 mmol scale, **General Procedure** was followed with acid **S9** (39.2 mg, 0.2 mmol, 1.0 equiv), vinyl iodide **S10** (97.8 mg, 0.3 mmol, 1.5 equiv), and triphenylphosphine (157.4 mg, 0.6 mmol, 3.0 equiv). Purification by preparative TLC (silica gel, Petroleum ether) afforded **64** (36 mg, 51%).

Physical state: colorless oil;

TLC: R$_f$ = 0.42 (silica gel, 19:1, Petroleum ether: EtOAc, UV);

^{1}H NMR (500 MHz, $CDCl_3$): δ 5.15 – 5.06 (m, 3H), 3.58 (t, J = 7.2 Hz, 2H), 2.27 – 2.19 (m, 2H), 2.12 – 2.02 (m, 4H), 2.02 – 1.94 (m, 4H), 1.68 (s, 3H), 1.64 – 1.58 (m, 9H), 0.89 (s, 9H), 0.05 (s, 6H);

^{13}C NMR (101 MHz, CDCl3): δ 137.2, 135.1, 131.4, 124.5, 124.3, 120.3, 63.3, 39.9, 39.9, 32.0, 31.8, 26.9, 26.7, 26.1, 25.9, 22.8, 18.5, 17.8, 16.3, 16.1, 14.3, –5.1.

HRMS (ESI): Calcd for $C_{22}H_{43}OSi$ [M+H]$^+$: 351.3078; found 351.3083.

References

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[3] S. J. Harwood, M. D. Palkowitz, C. N. Gannett, P. Perez, Z. Yao, L. Sun, H. D. Abruña, S. L. Anderson and P. S. Baran, Modular Terpene Synthesis enabled by Mild Electrochemical Couplings, *Science*, 2022, **375**, 745–752.

[4] B. Jin, Q. Liu and G. A. Sulikowski, Development of an end-game strategy towards apoptolidin: a sequential Suzuki coupling approach, *Tetrahedron*, 2005, **61**, 401–408.

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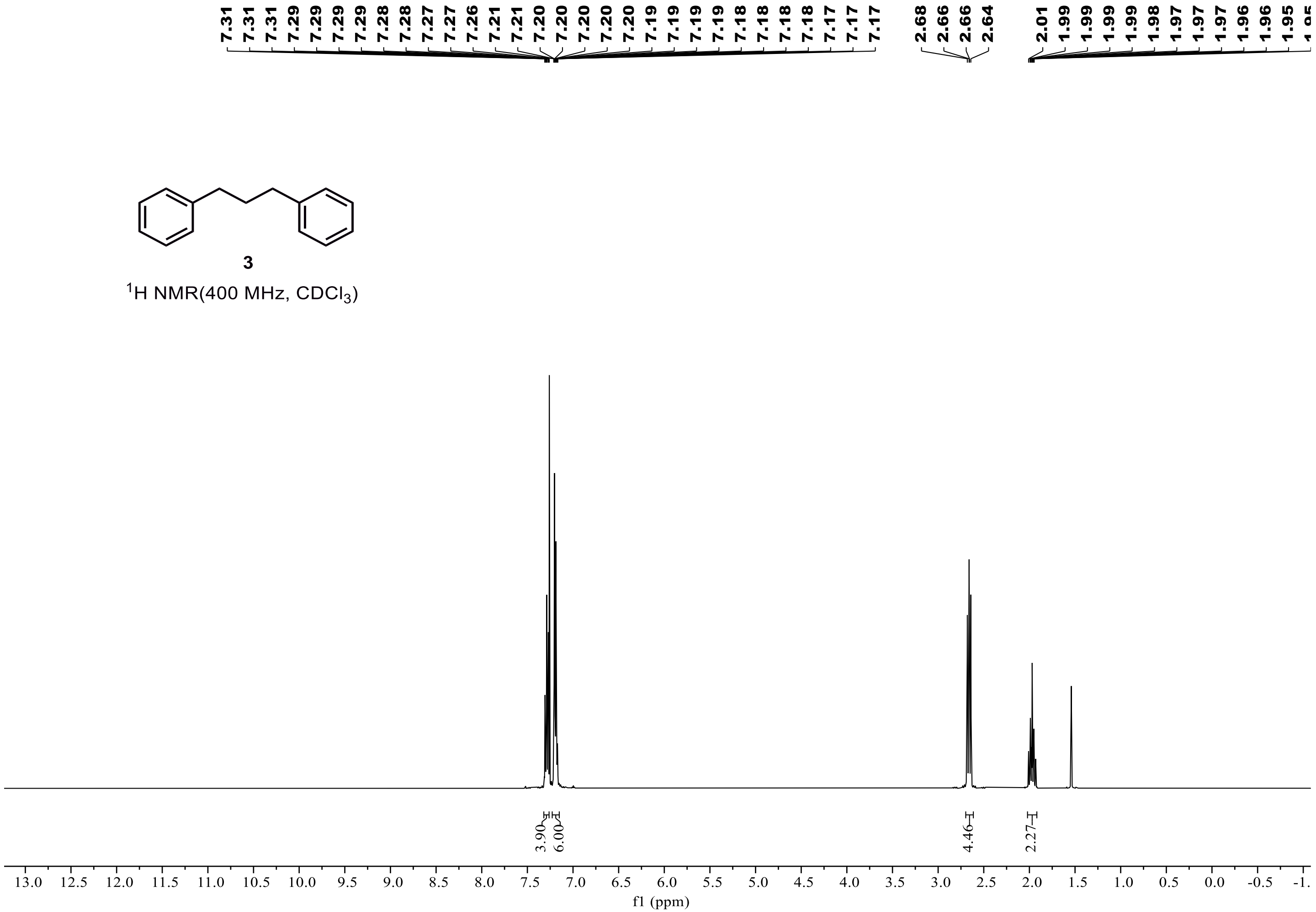
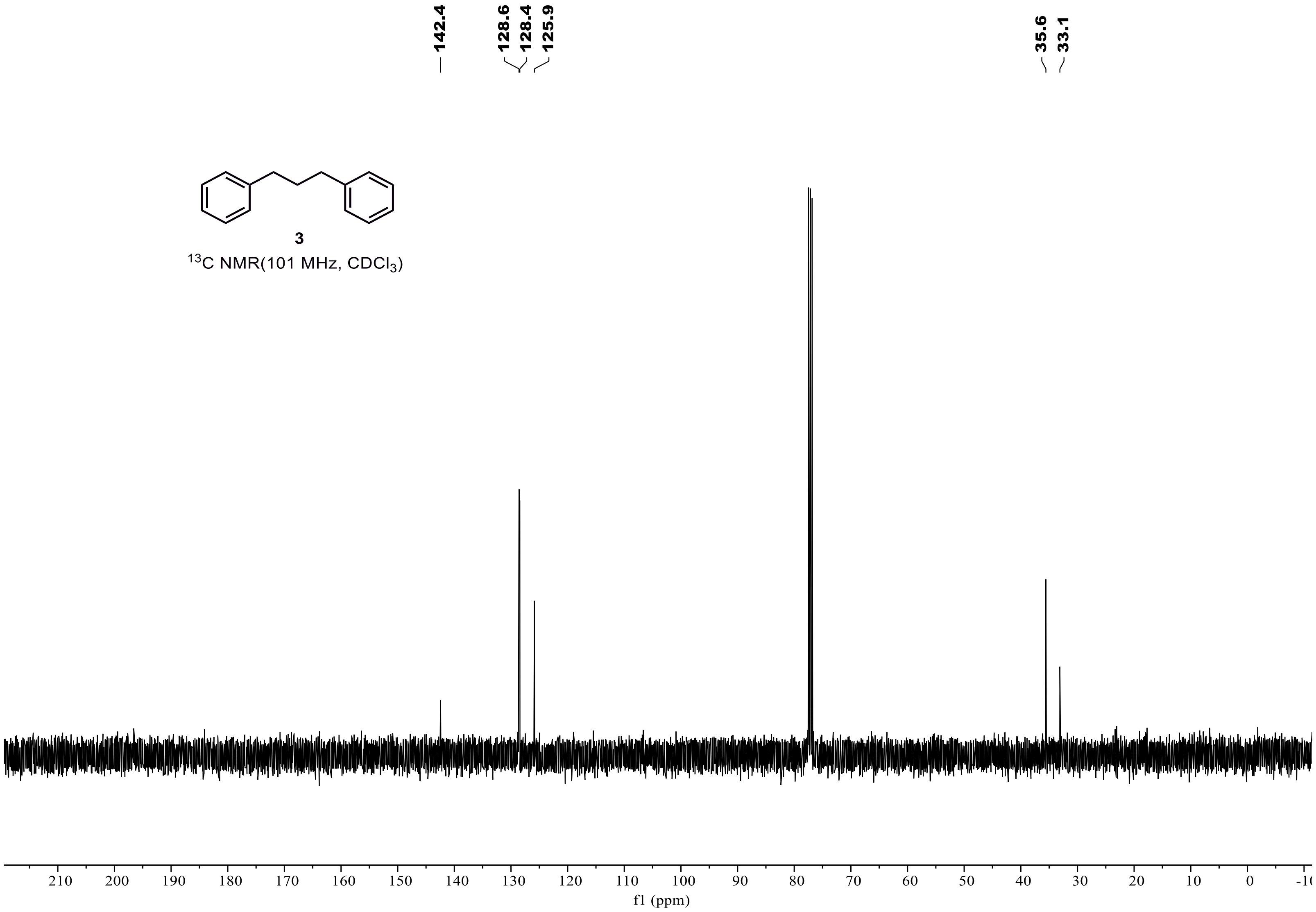
NMR Spectra



3

^{1}H NMR(400 MHz, CDCl$_3$)


142.4
128.6
128.4
125.9
35.6
33.1
3
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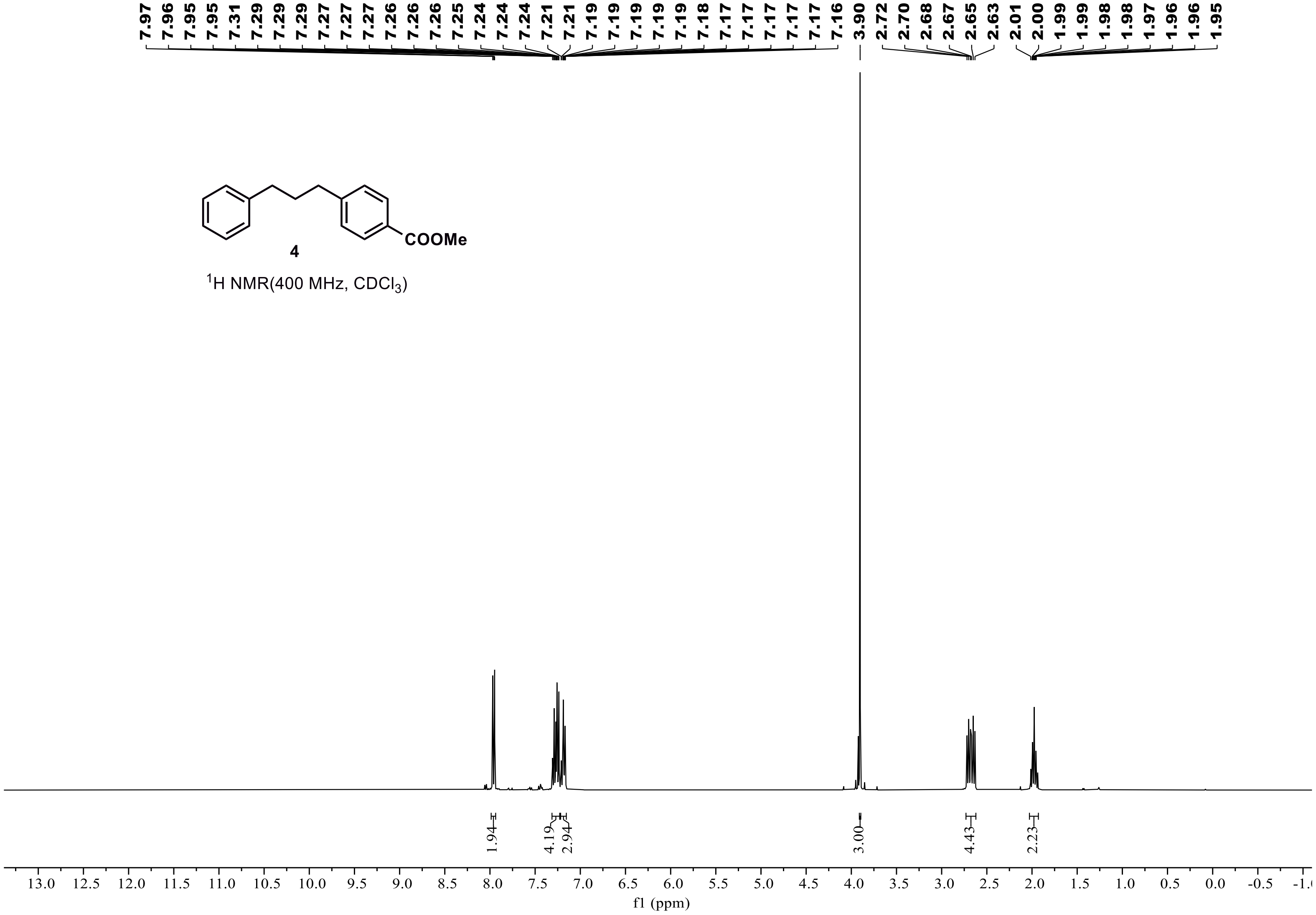
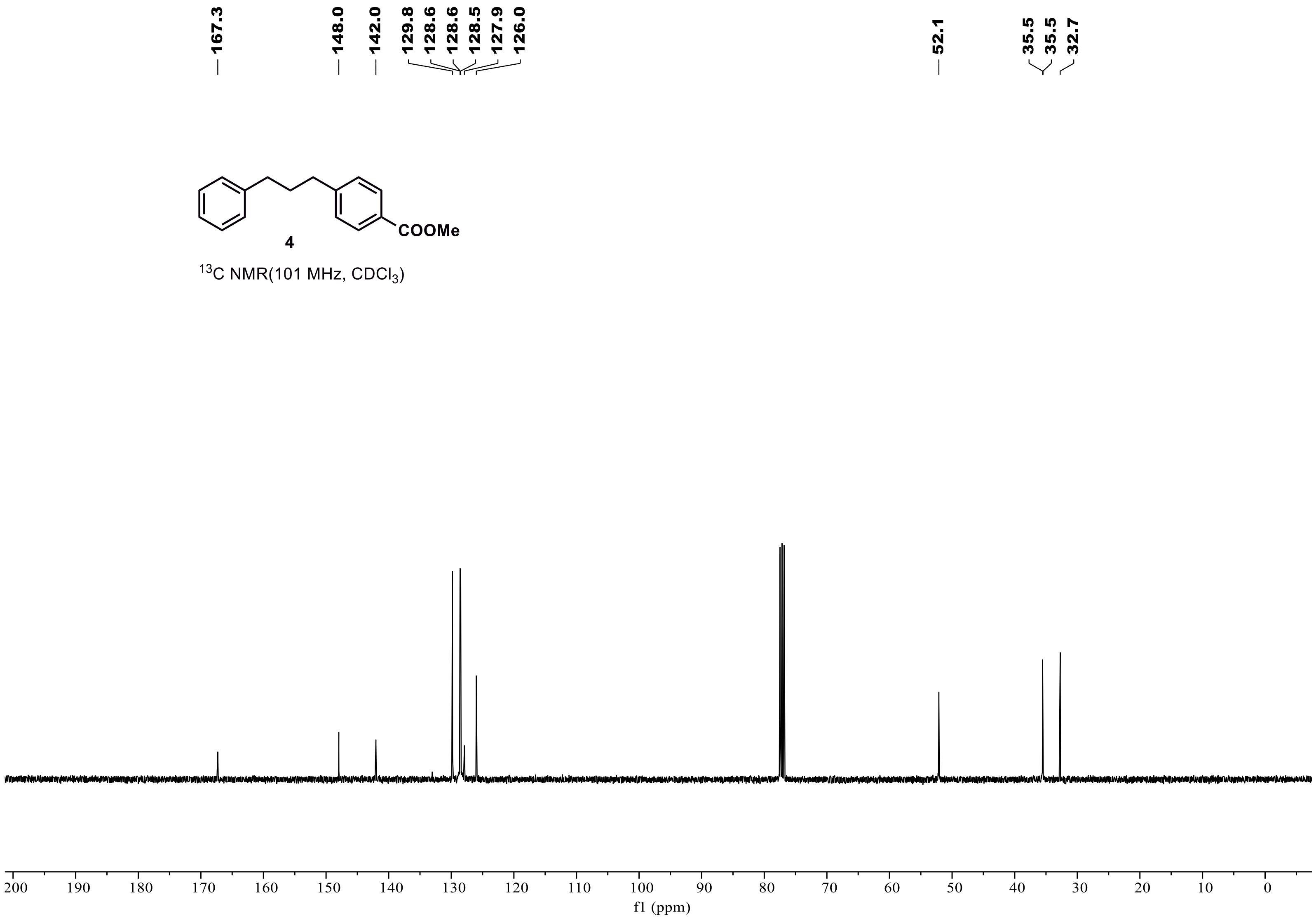
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



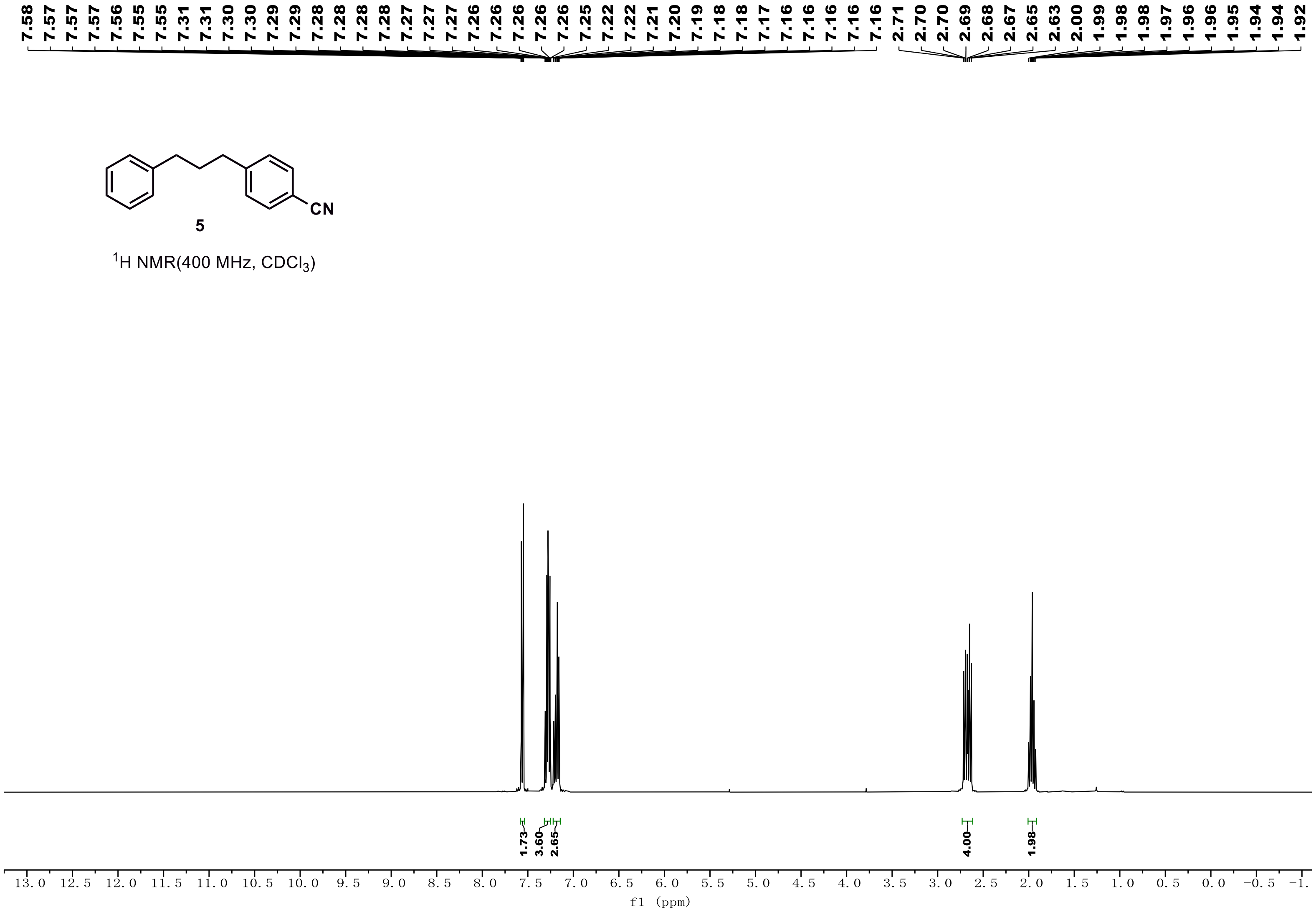
4

^{1}H NMR(400 MHz, $CDCl_3$)

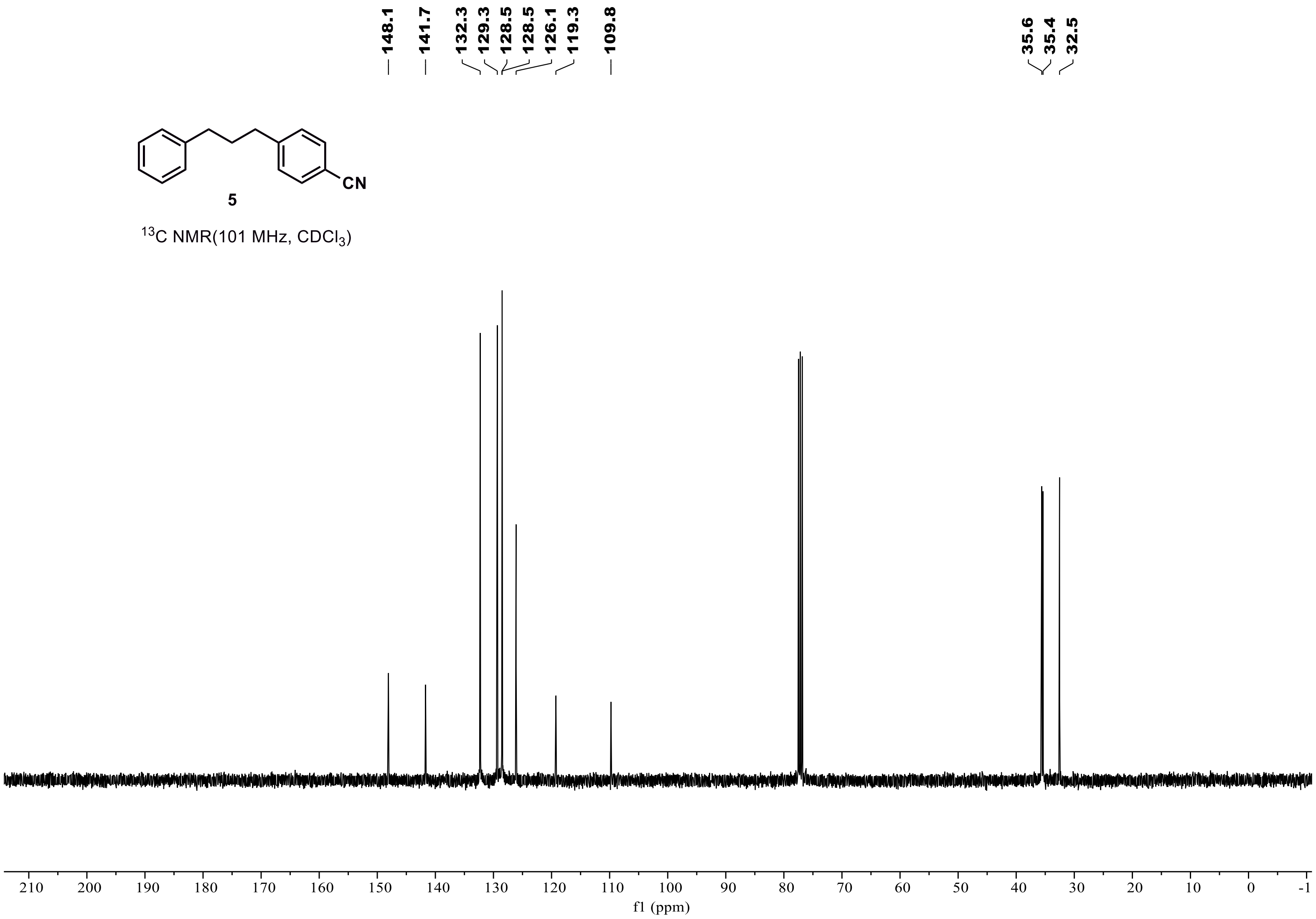


4

^{13}C NMR(101 MHz, $CDCl_3$)

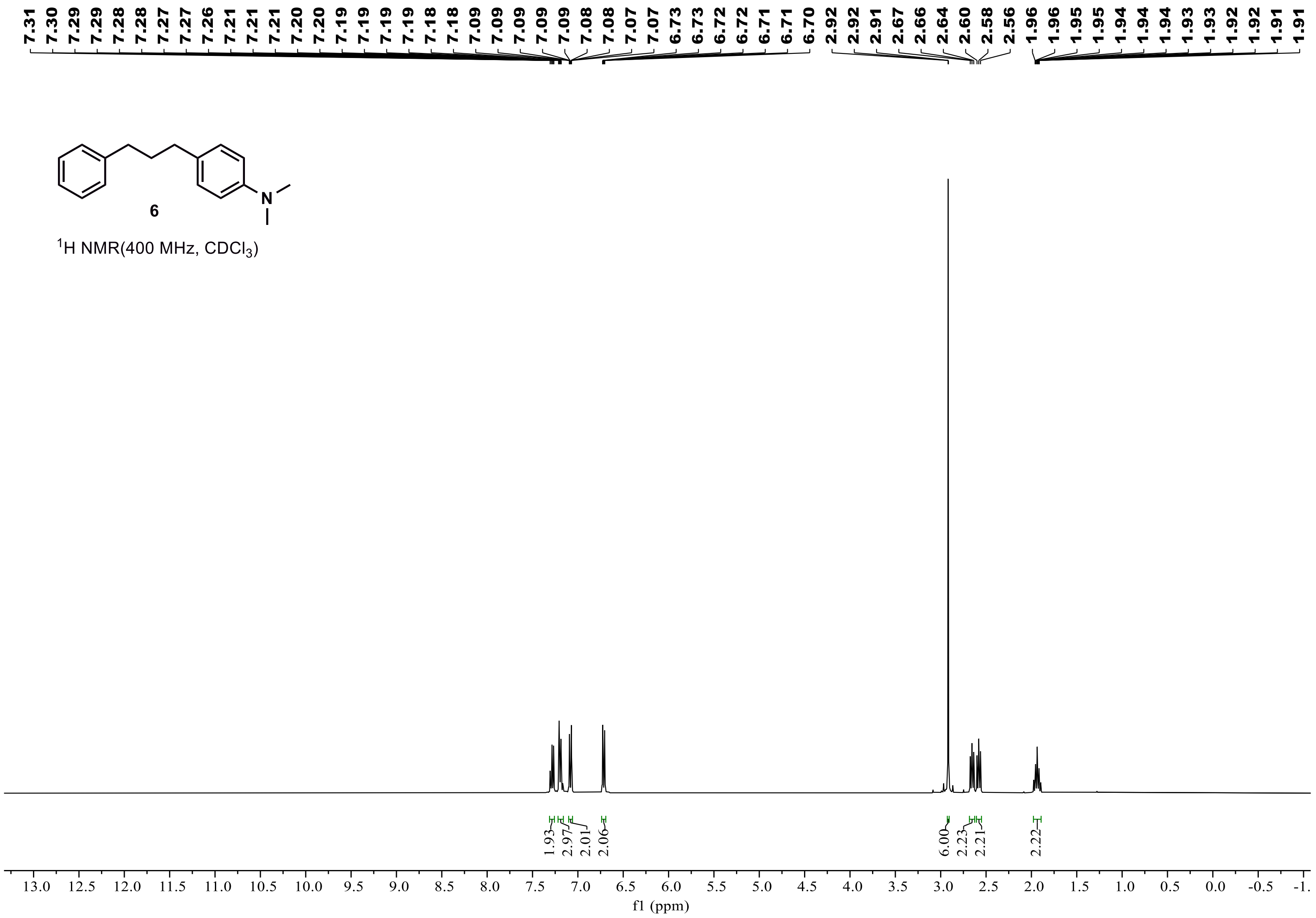

5

^{1}H NMR(400 MHz, CDCl$_3$)



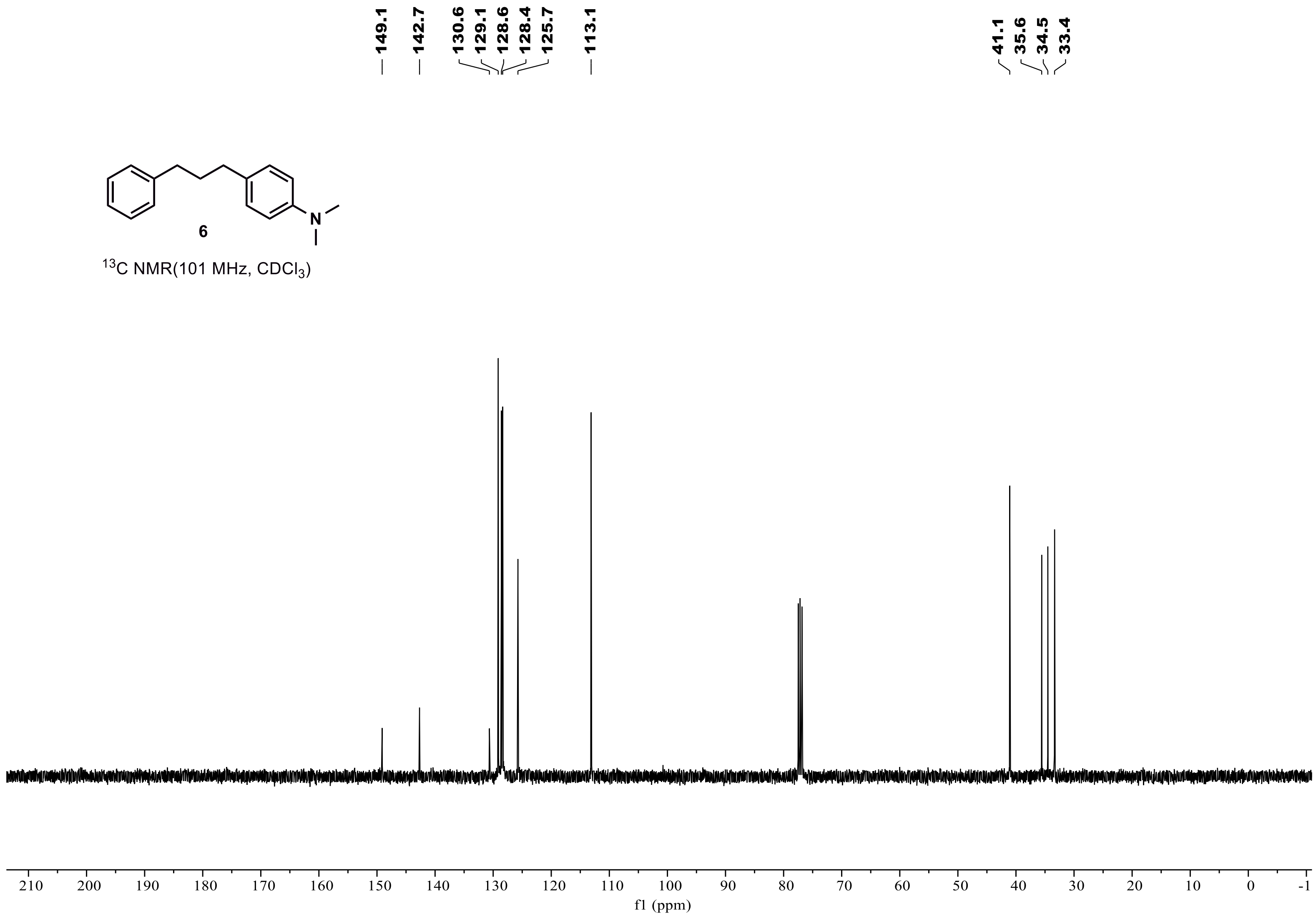
5

^{13}C NMR(101 MHz, $CDCl_3$)



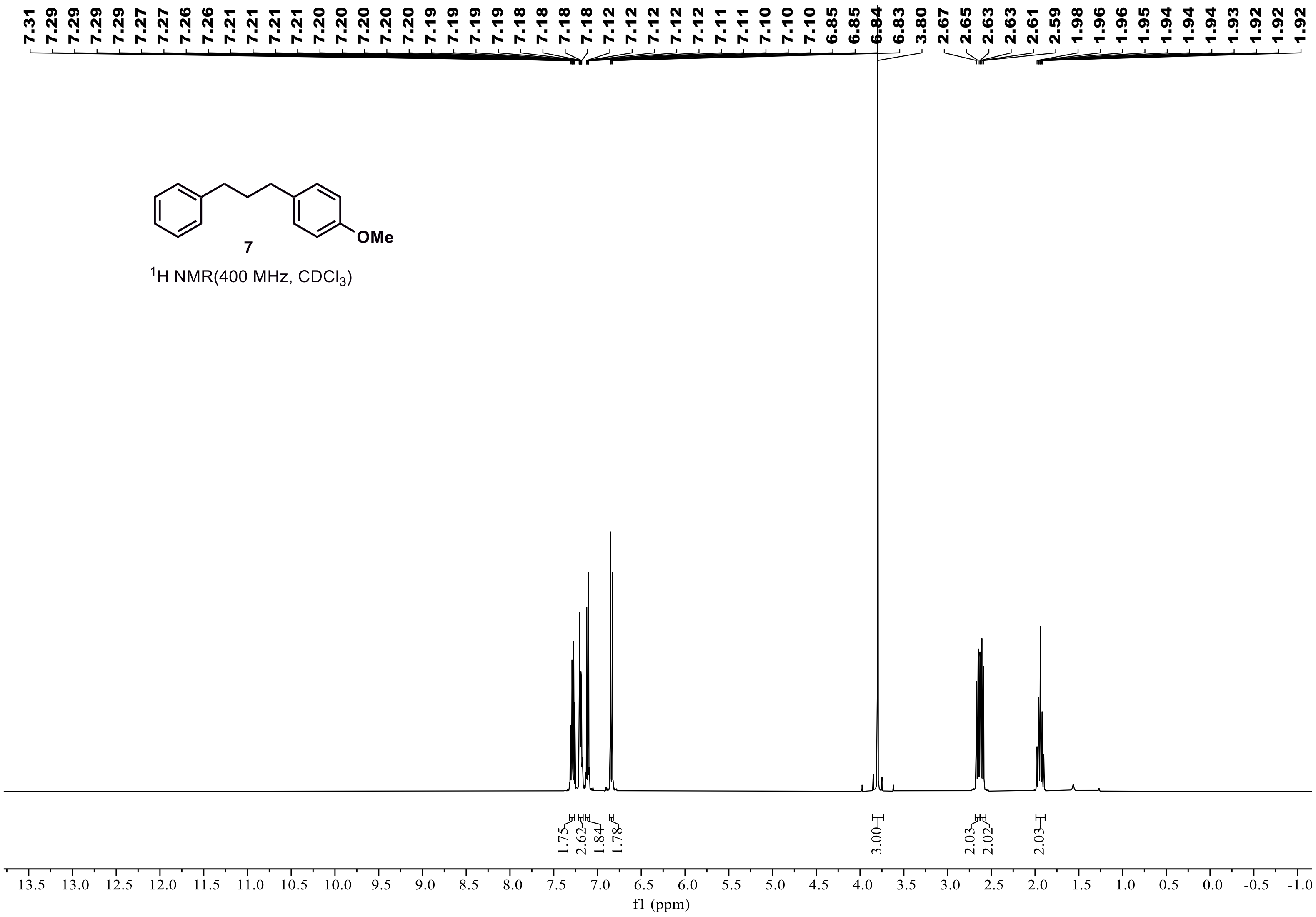
6

^{1}H NMR(400 MHz, CDCl$_3$)



6

^{13}C NMR(101 MHz, CDCl$_3$)


7.31
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2.59
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1.94
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1.92
OMe
7
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2.62
1.84
1.78
3.00
2.03
2.02
2.03
13.5
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12.5
12.0
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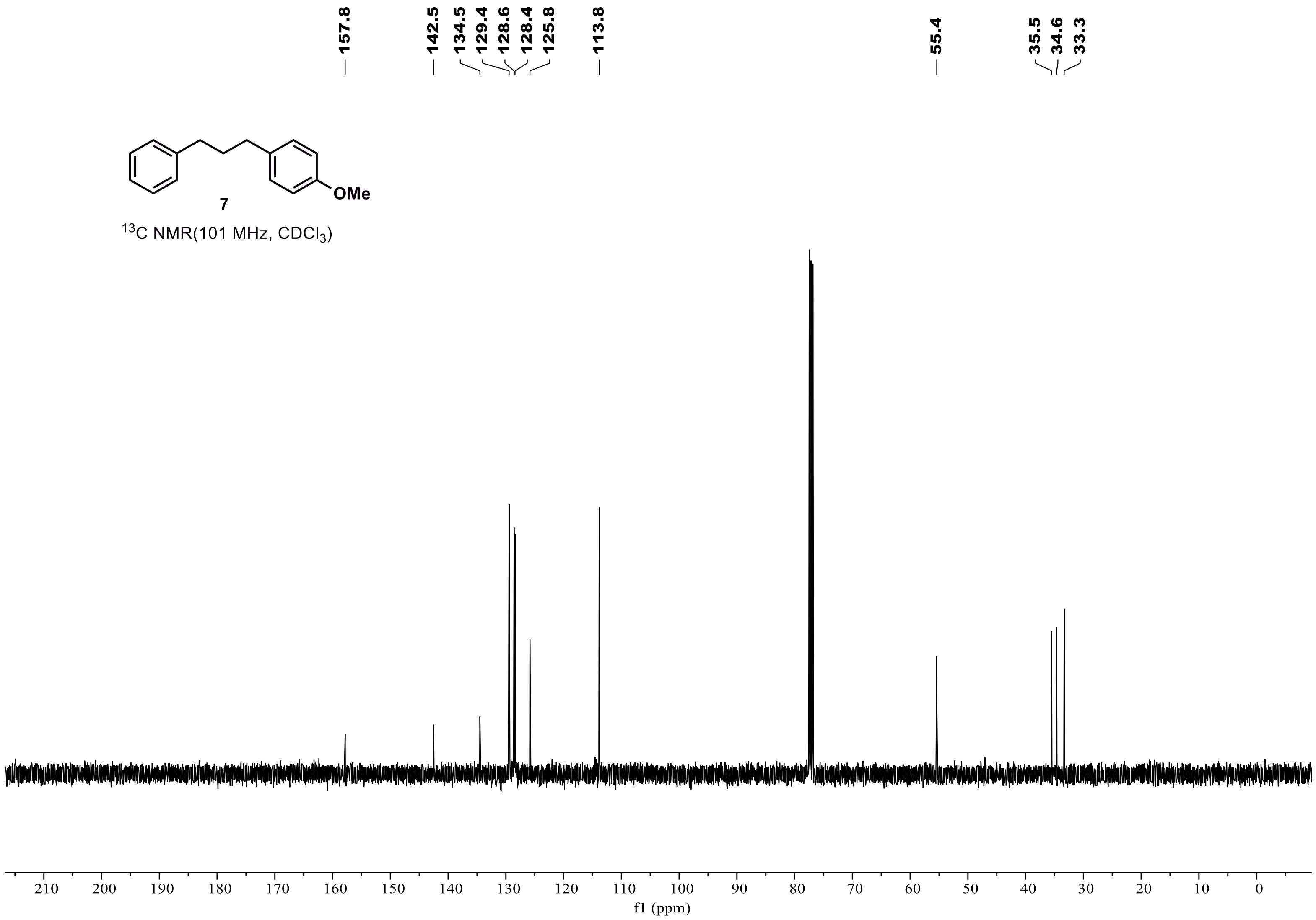
0.0
-0.5
-1.0
f1 (ppm)


157.8
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134.5
129.4
128.6
128.4
125.8
113.8
55.4
35.5
34.6
33.3
OMe
7
^{13}C NMR(101 MHz, CDCl$_3$)
210
200
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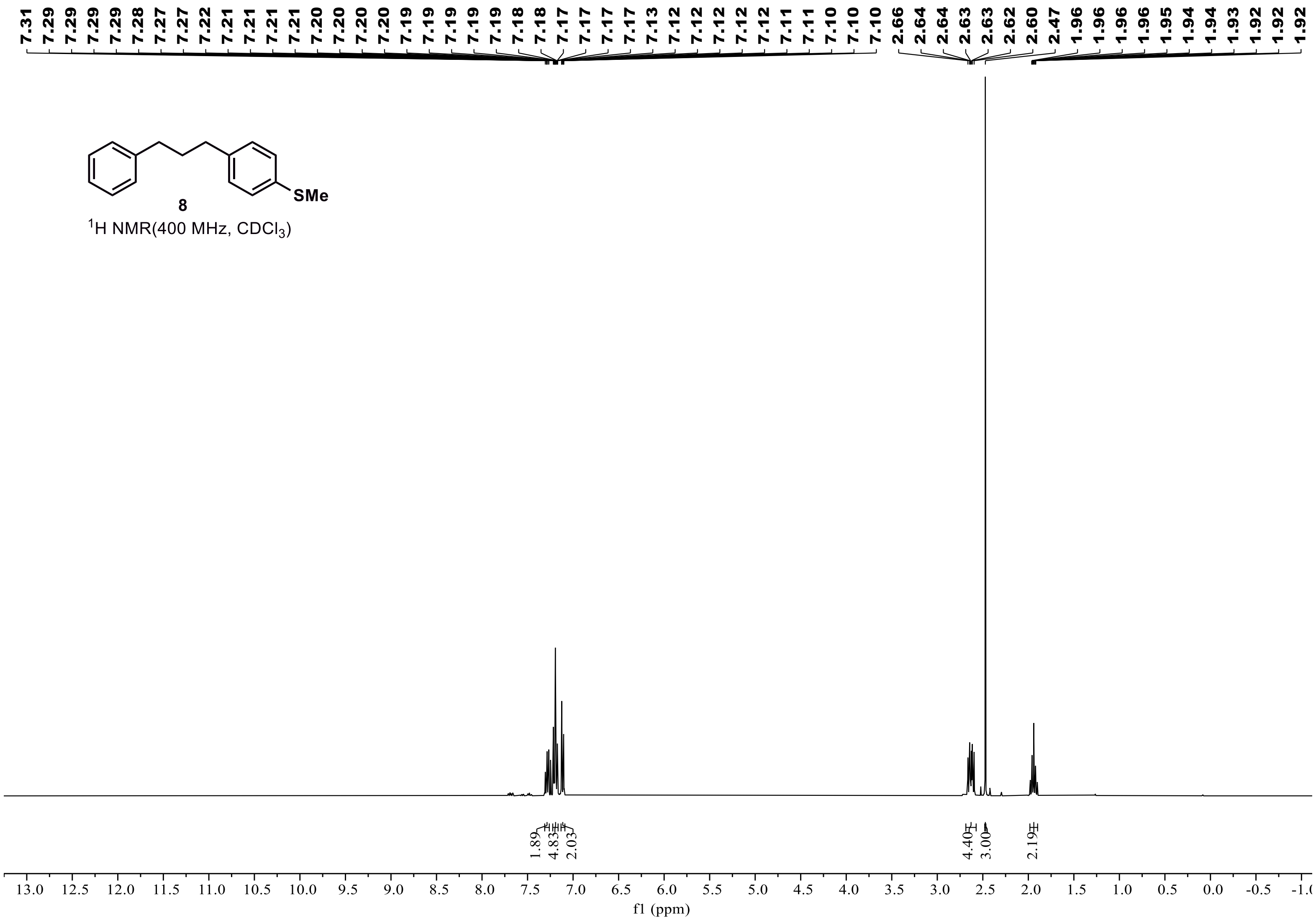
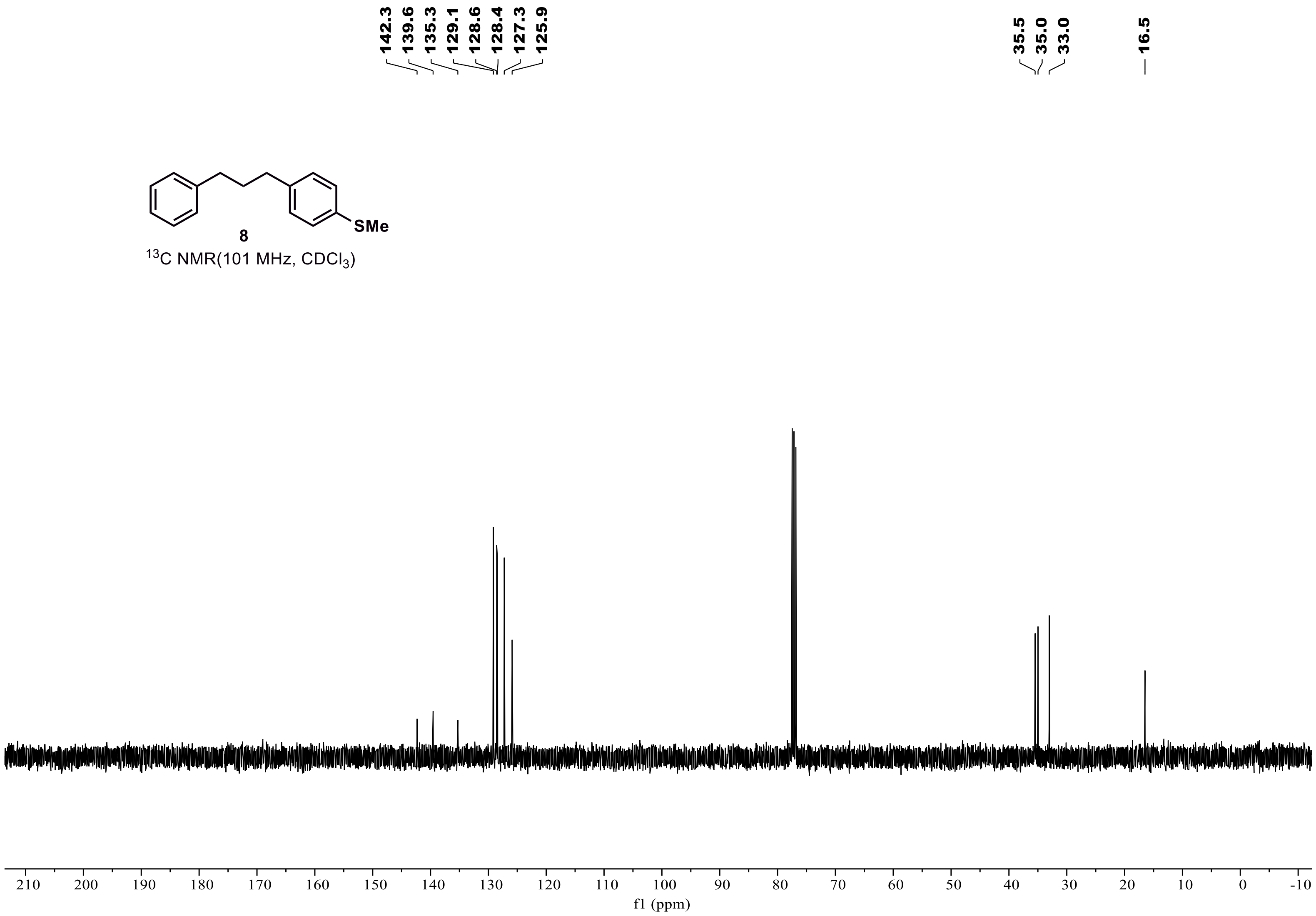
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



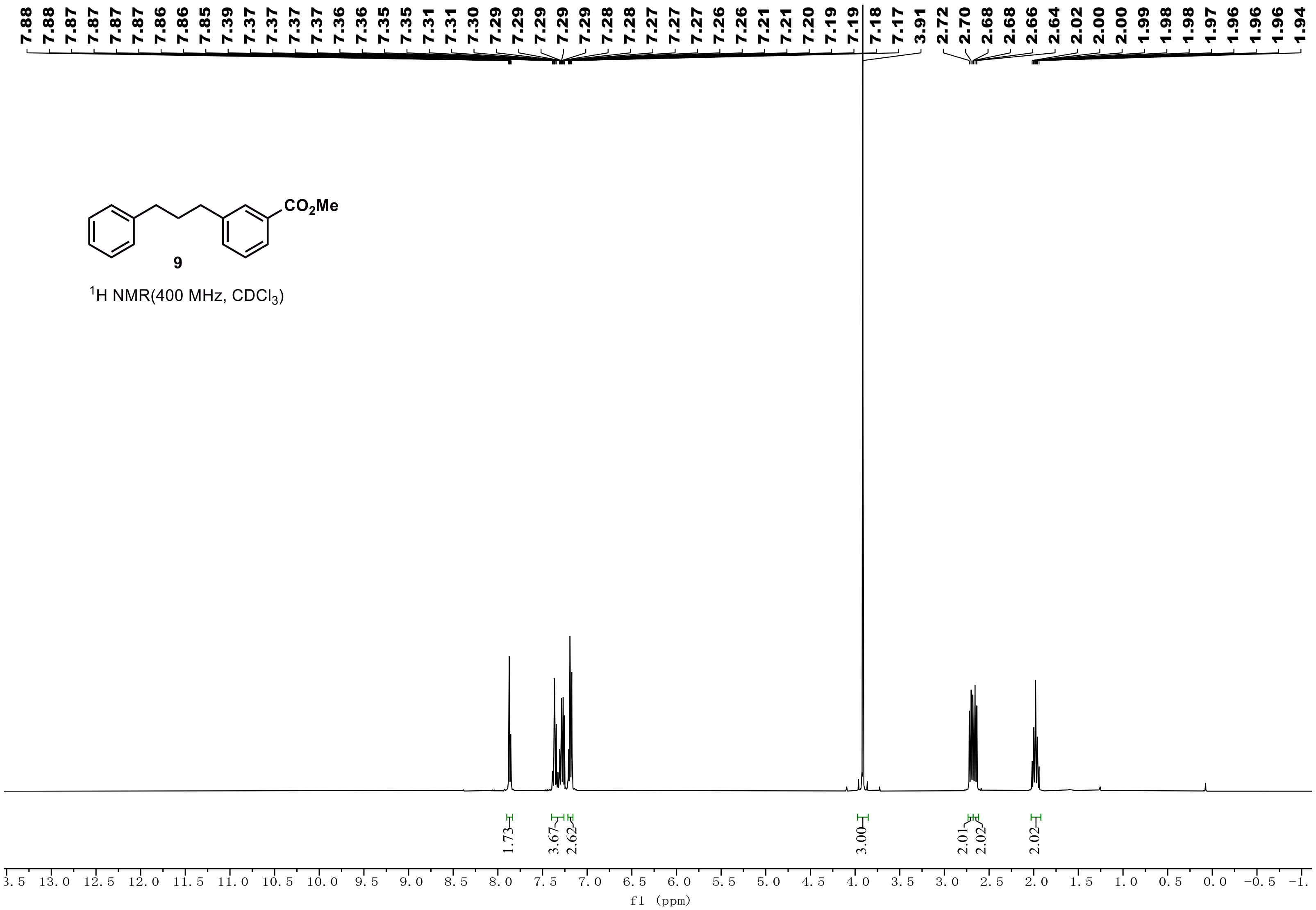
8

^{1}H NMR(400 MHz, CDCl$_3$)



8

^{13}C NMR(101 MHz, $CDCl_3$)


7.88
7.88
7.87
7.87
7.87
7.87
7.86
7.86
7.85
7.39
7.37
7.37
7.37
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7.26
7.21
7.21
7.20
7.19
7.19
7.18
7.17
3.91
2.72
2.70
2.68
2.68
2.66
2.64
2.02
2.00
2.00
1.99
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1.98
1.97
1.96
1.96
1.96
1.94
CO2Me
9
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1.73
3.67
2.62
3.00
2.01
2.02
2.02
3.5
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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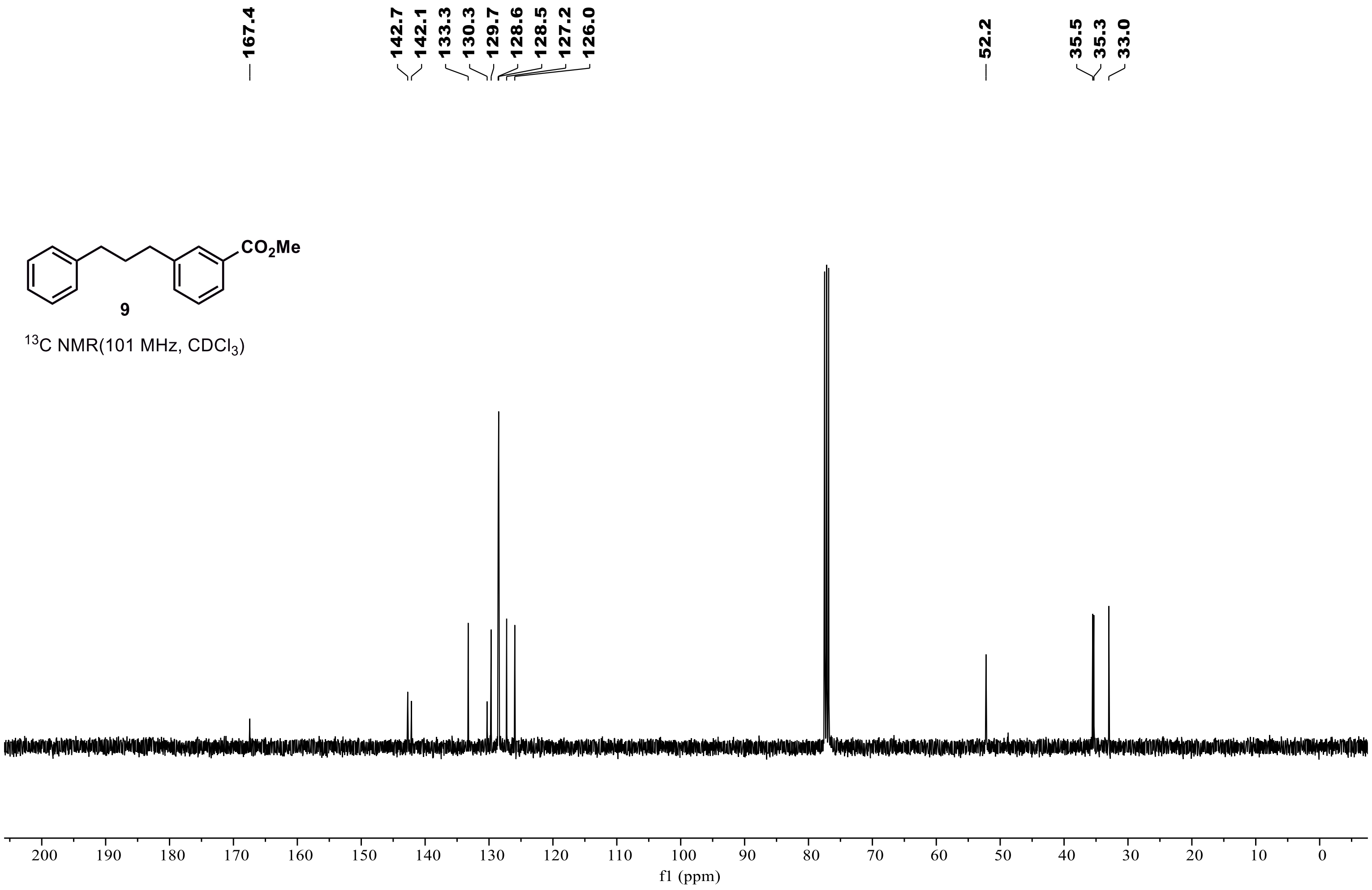
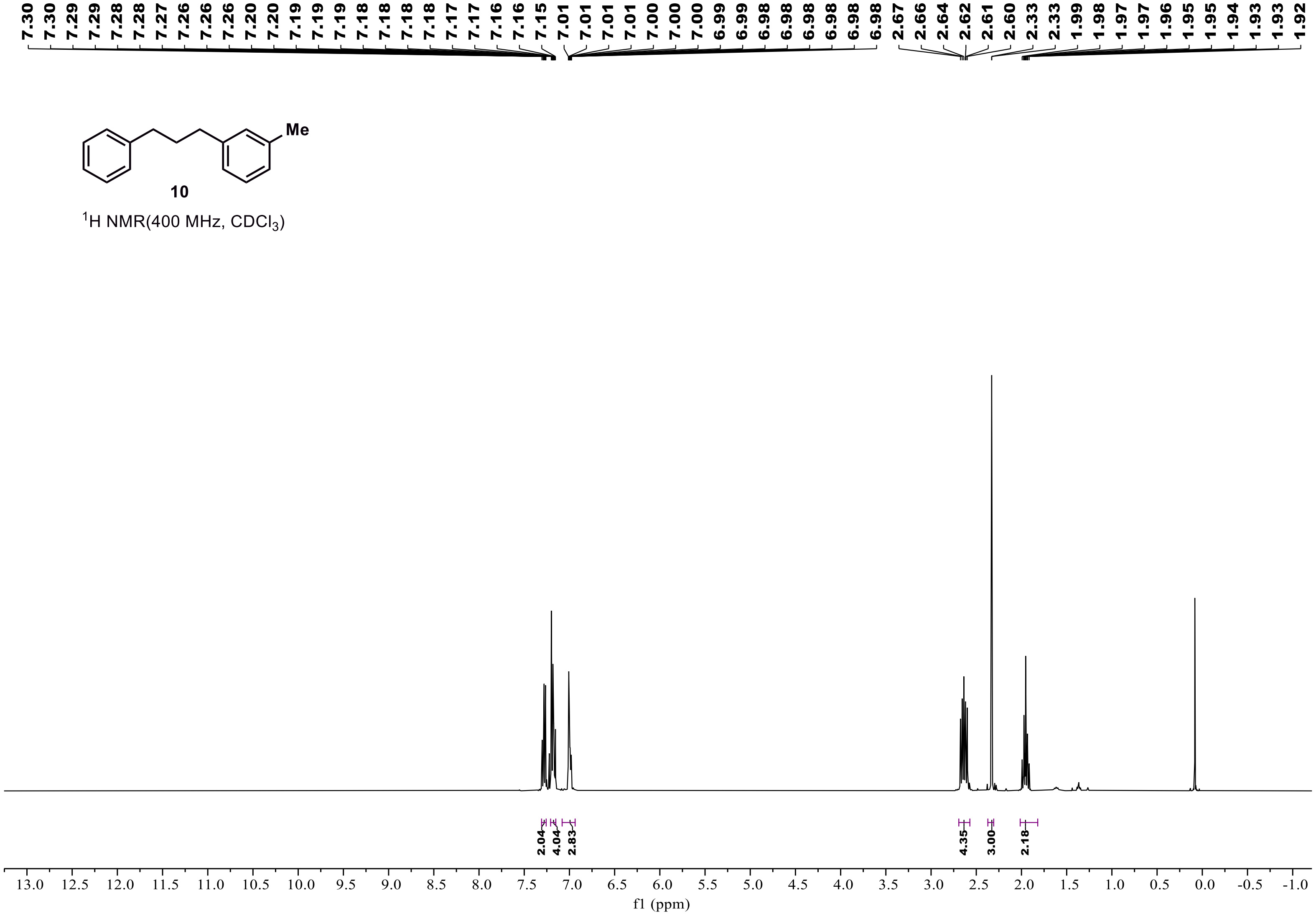
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.
f1 (ppm)



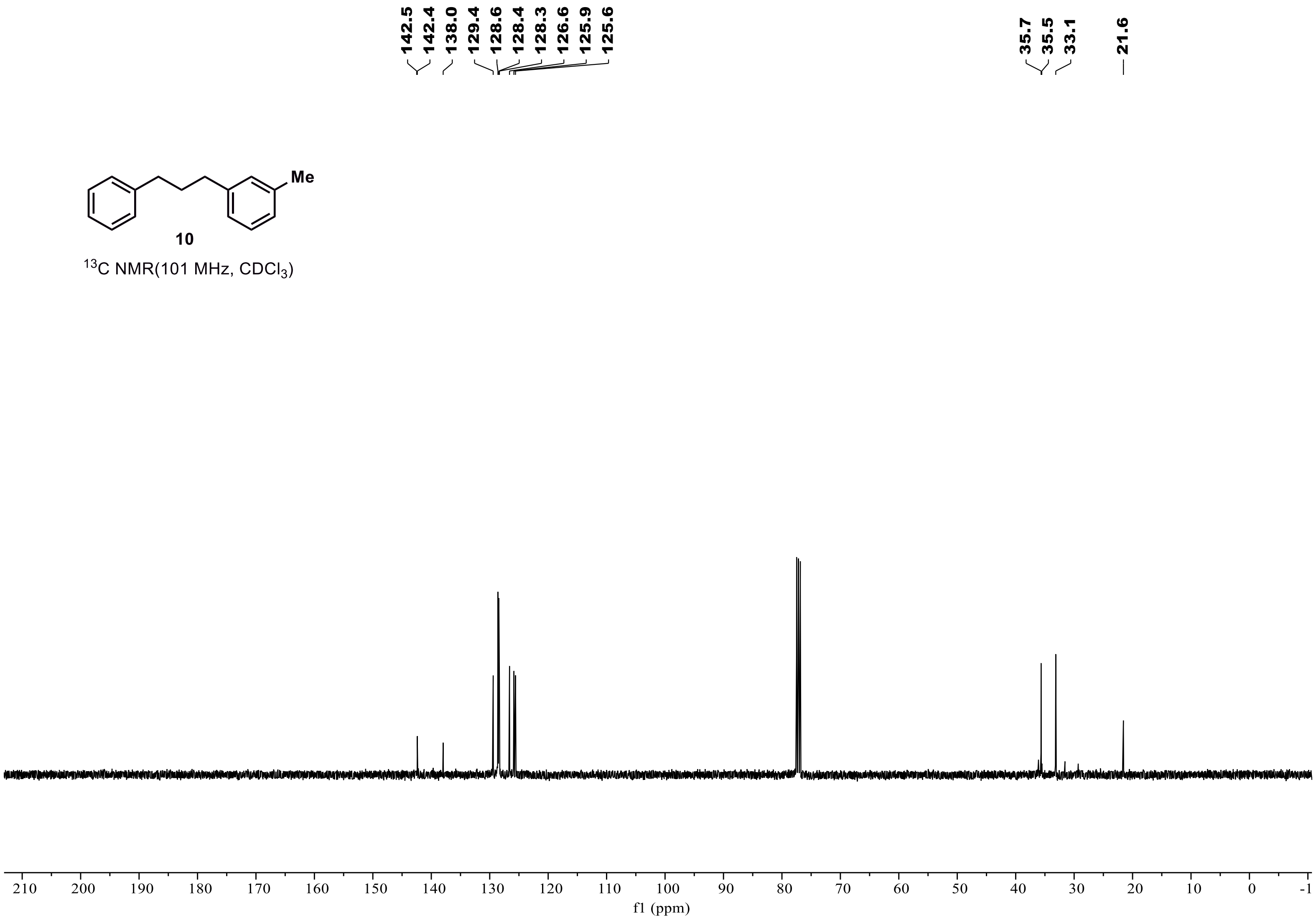
9

^{13}C NMR(101 MHz, CDCl$_3$)



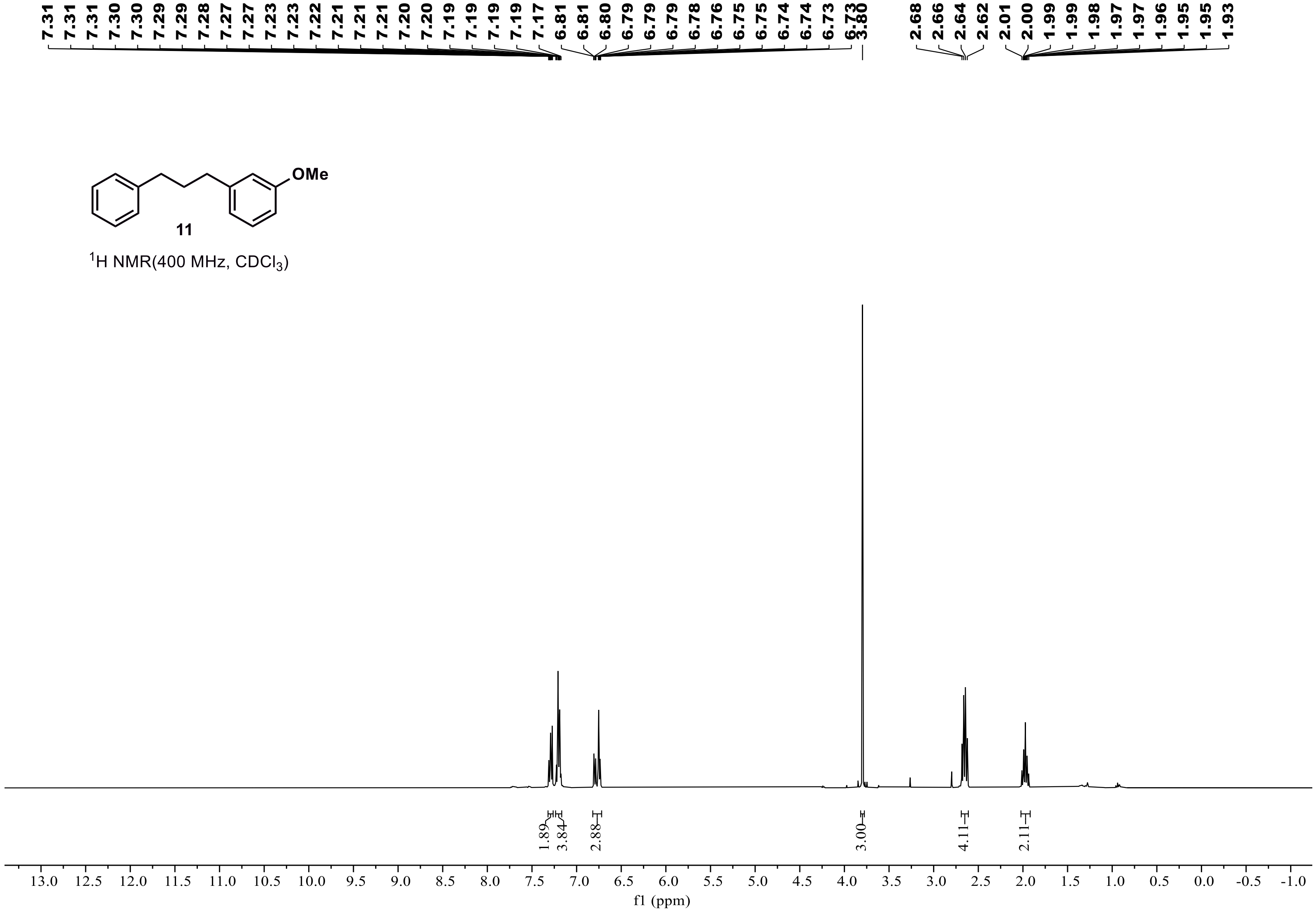
10

^{1}H NMR(400 MHz, CDCl$_3$)



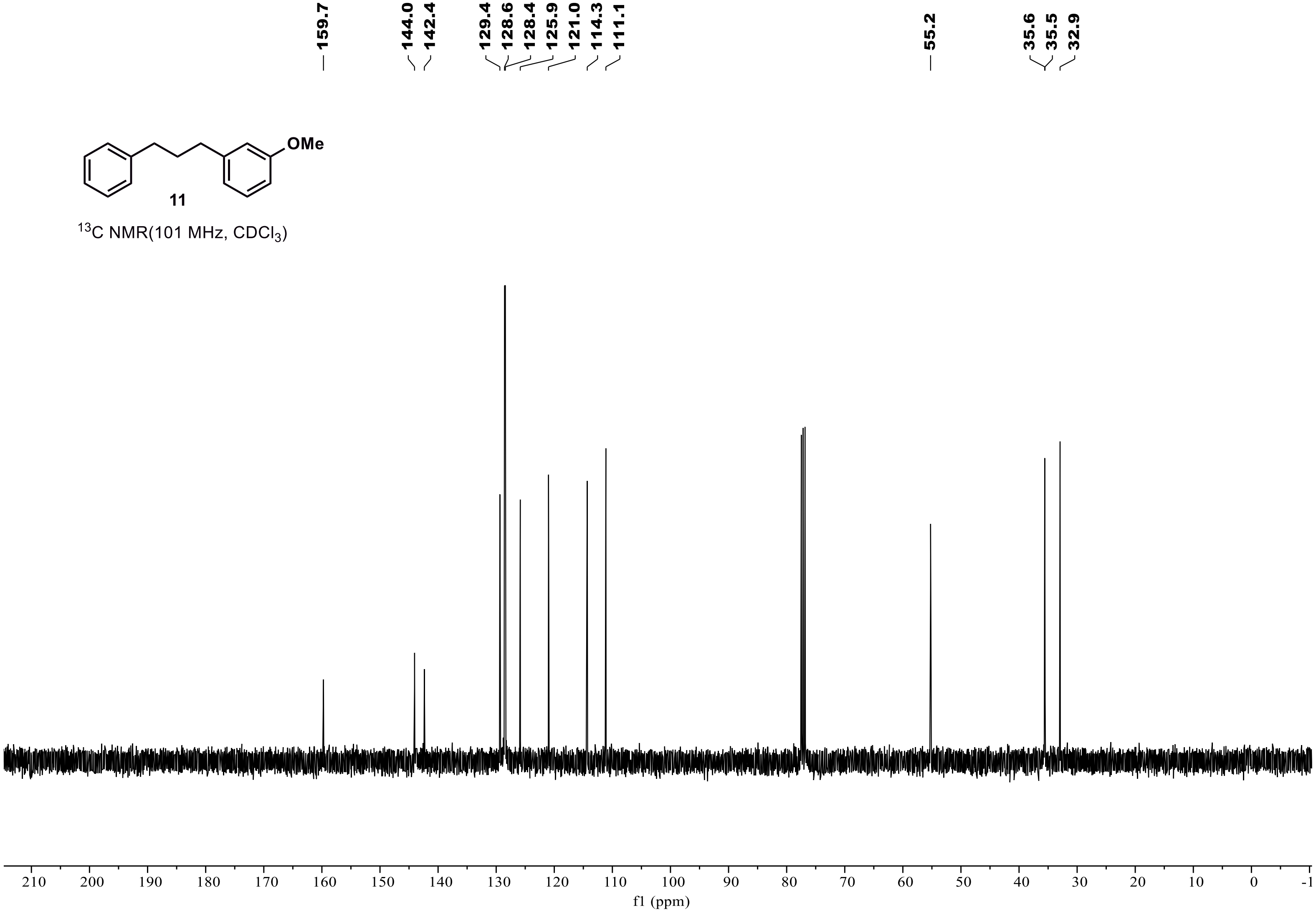
10

^{13}C NMR(101 MHz, $CDCl_3$)



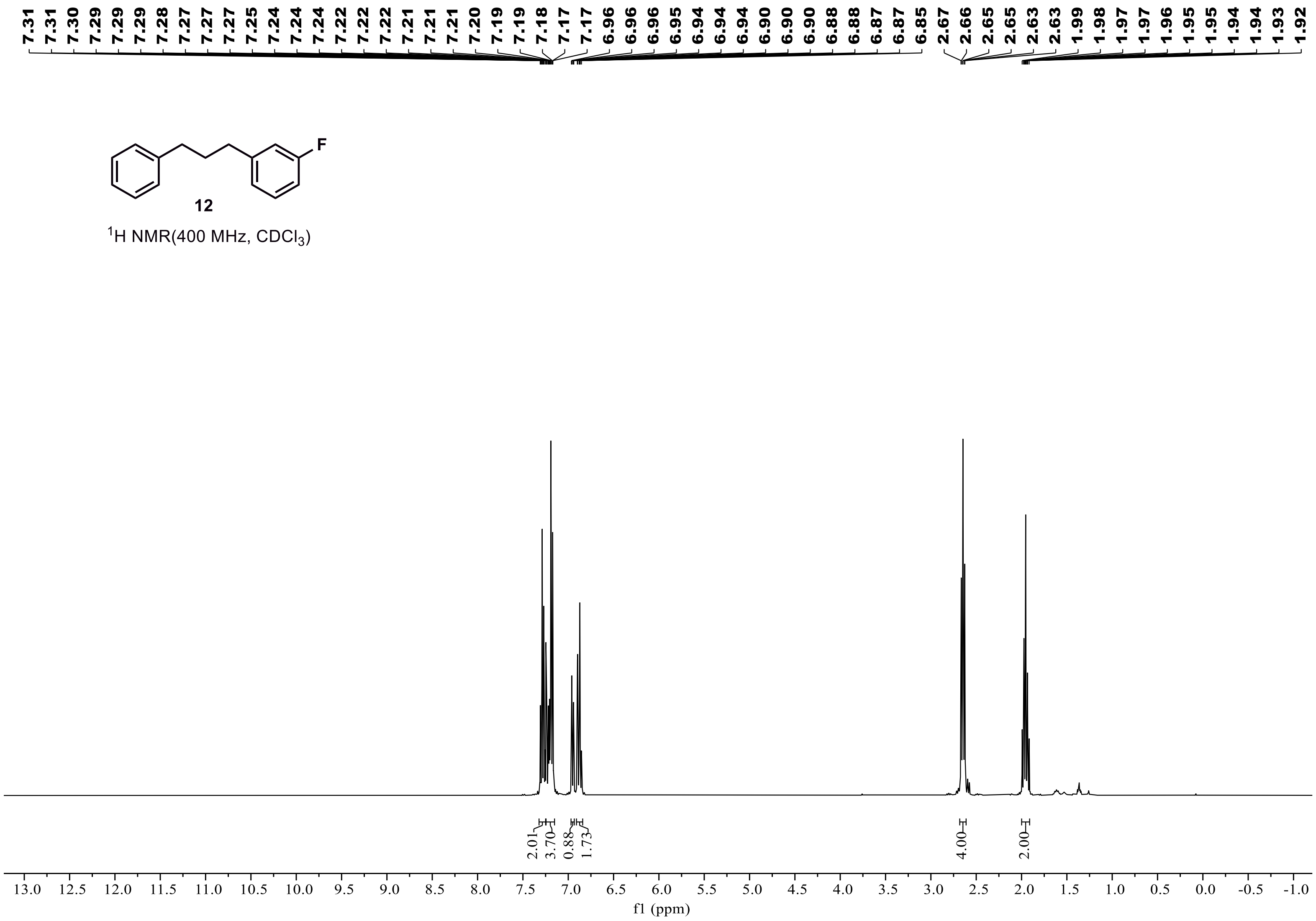
11

^{1}H NMR(400 MHz, $CDCl_3$)



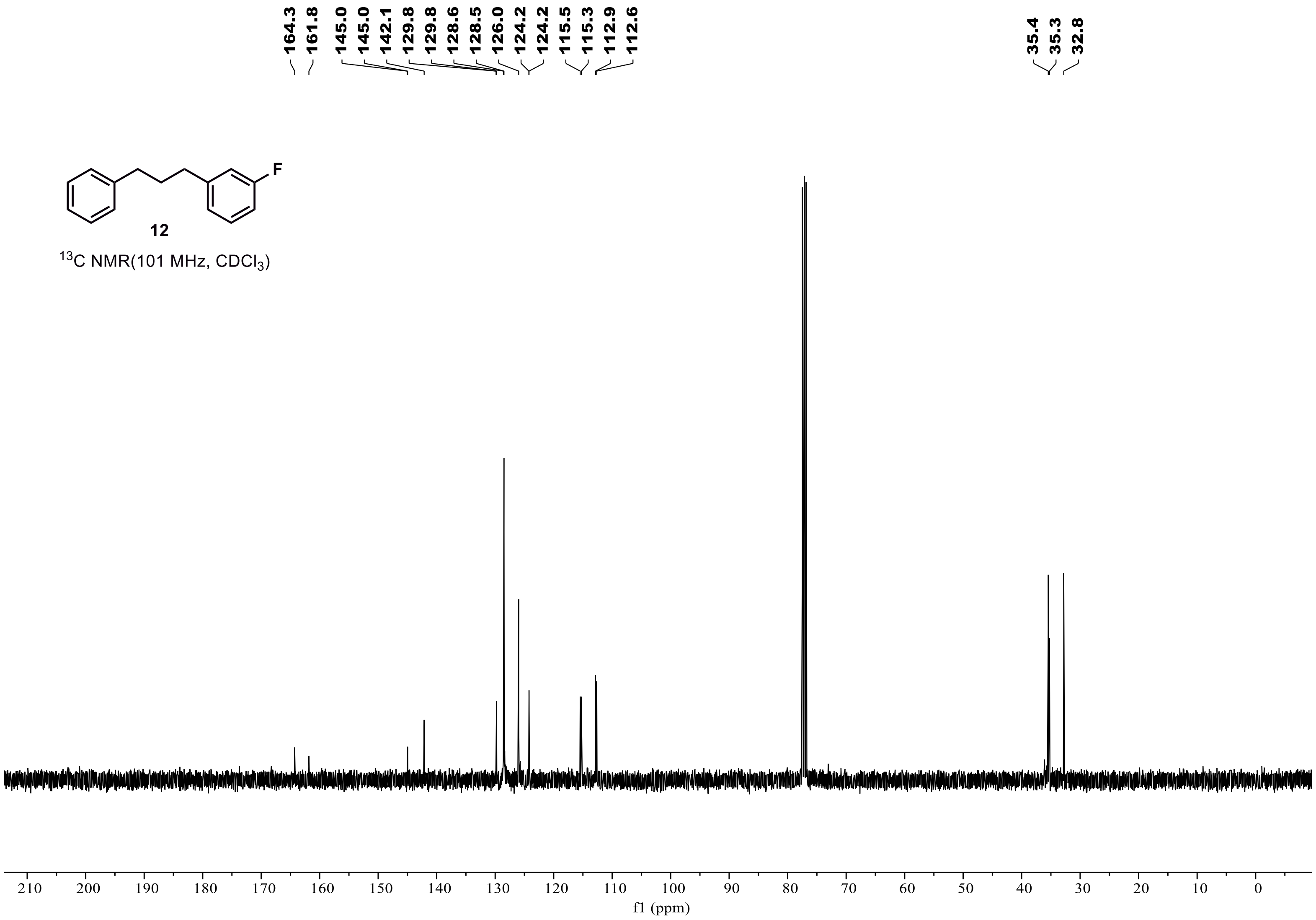
11

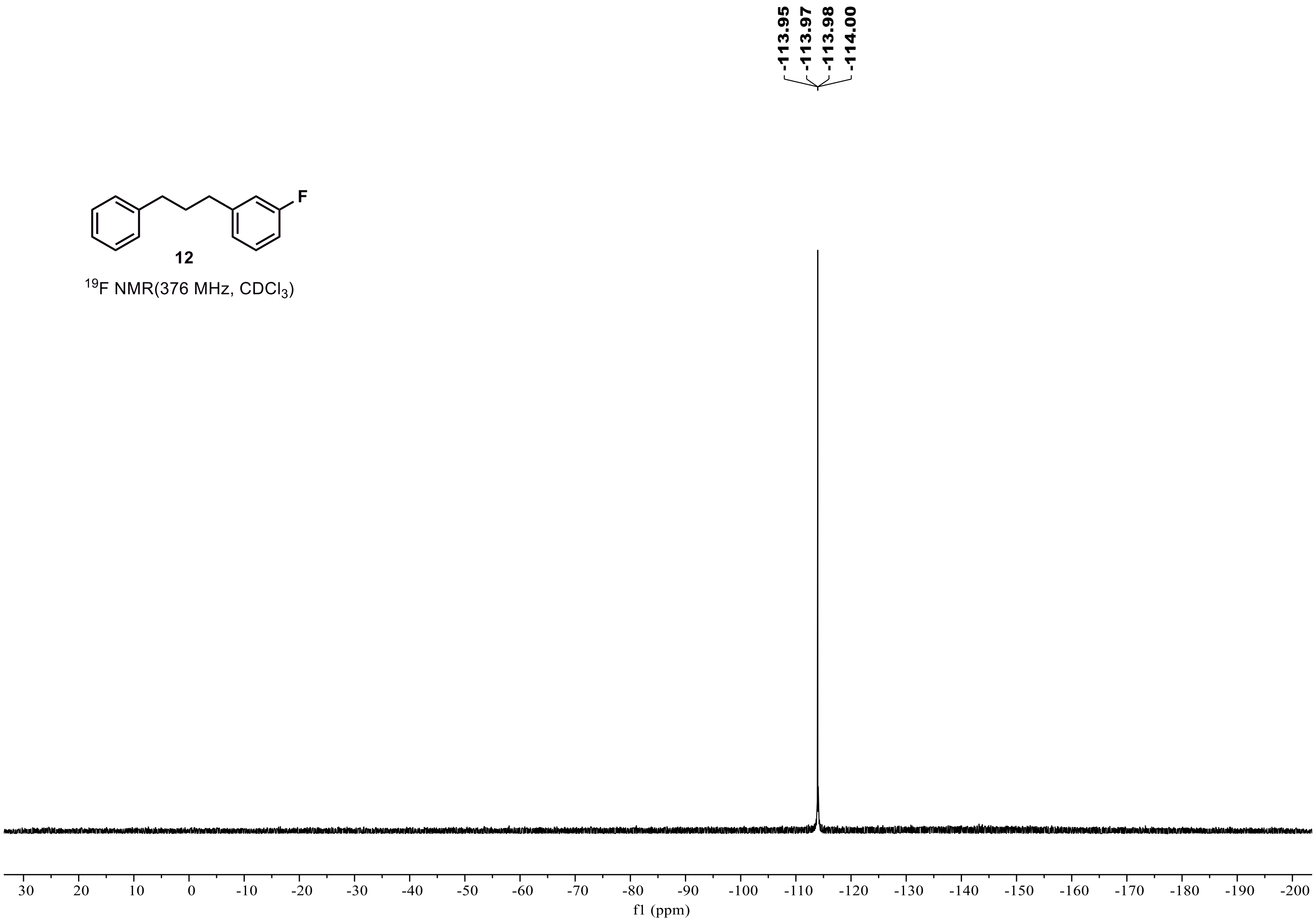
^{13}C NMR(101 MHz, CDCl$_3$)



12

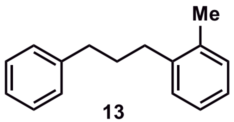
^{1}H NMR(400 MHz, $CDCl_3$)


164.3
161.8
145.0
145.0
142.1
129.8
129.8
128.6
128.5
126.0
124.2
124.2
115.5
115.3
112.9
112.6
35.4
35.3
32.8
F
12
^{13}C NMR(101 MHz, CDCl$_3$)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



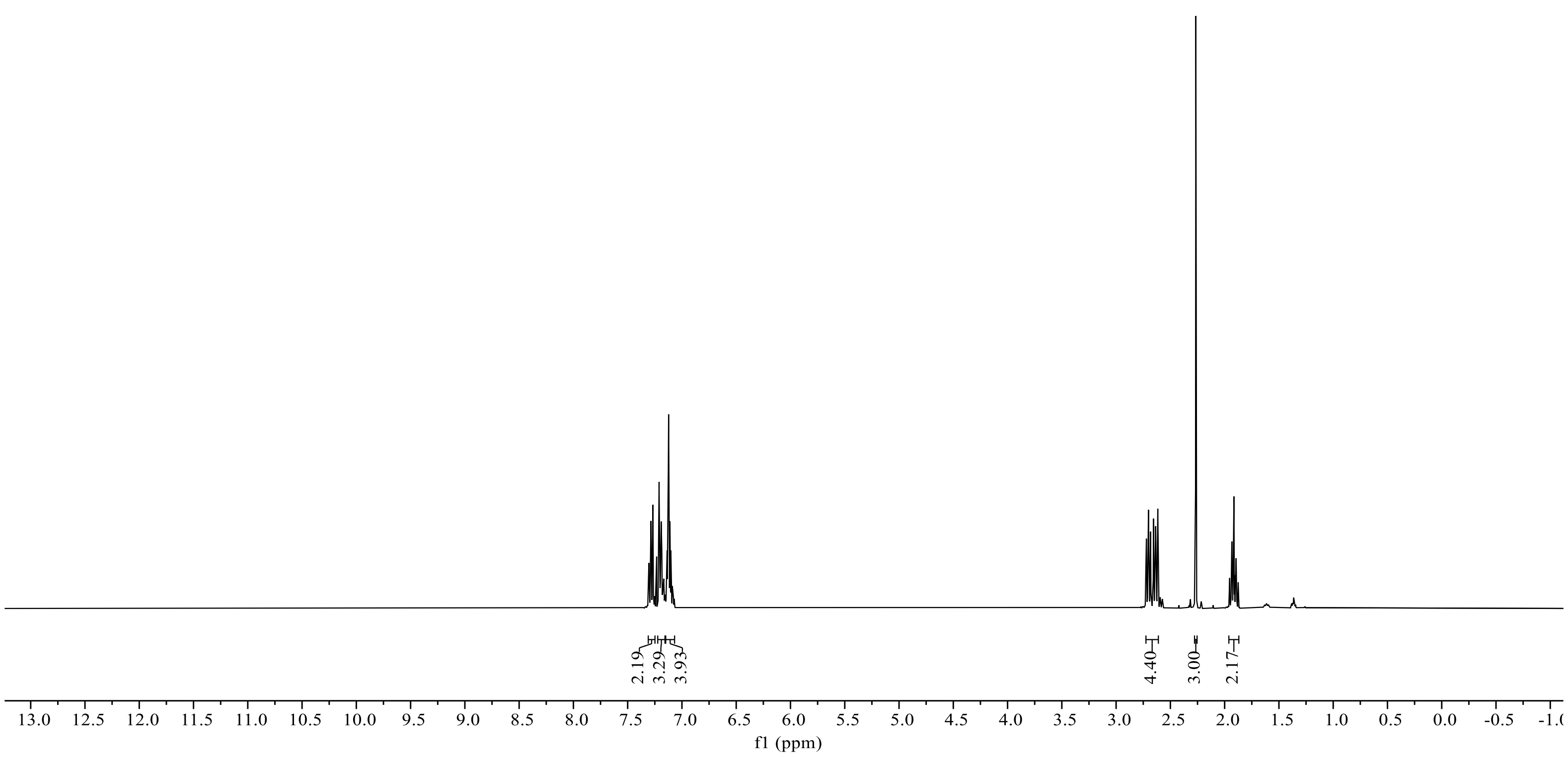
12

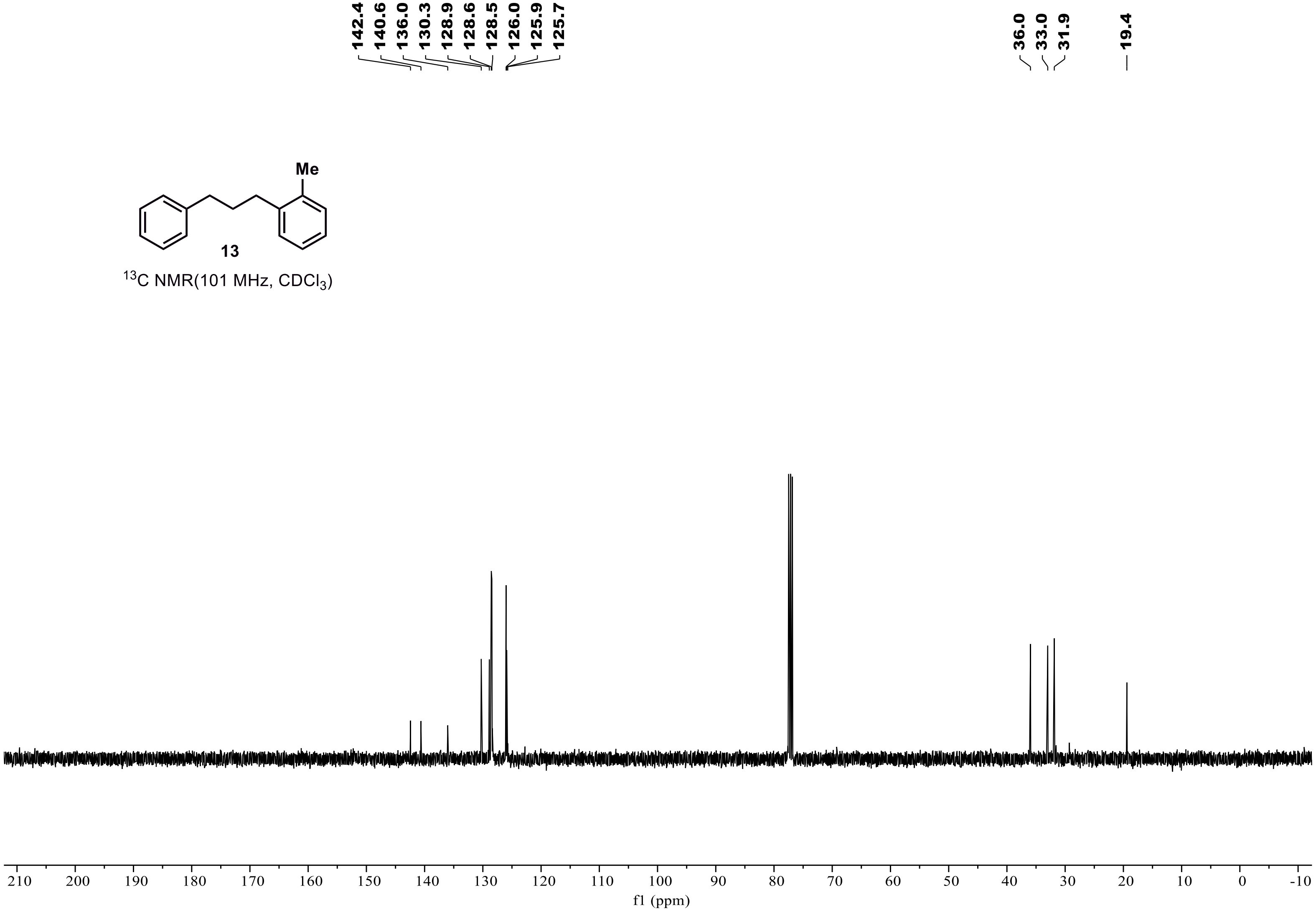
^{19}F NMR(376 MHz, $CDCl_3$)



13

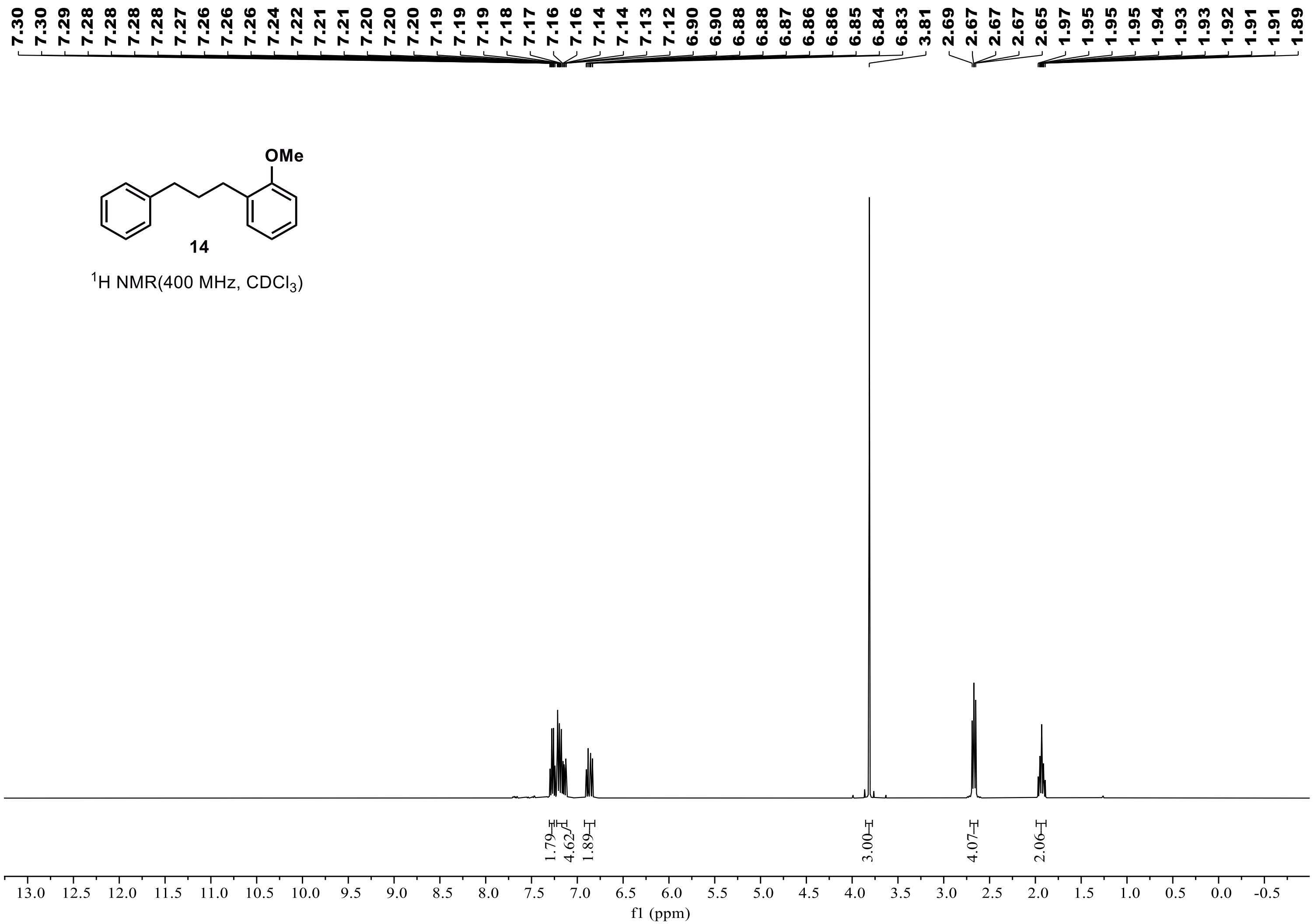
^{1}H NMR(400 MHz, $CDCl_3$)

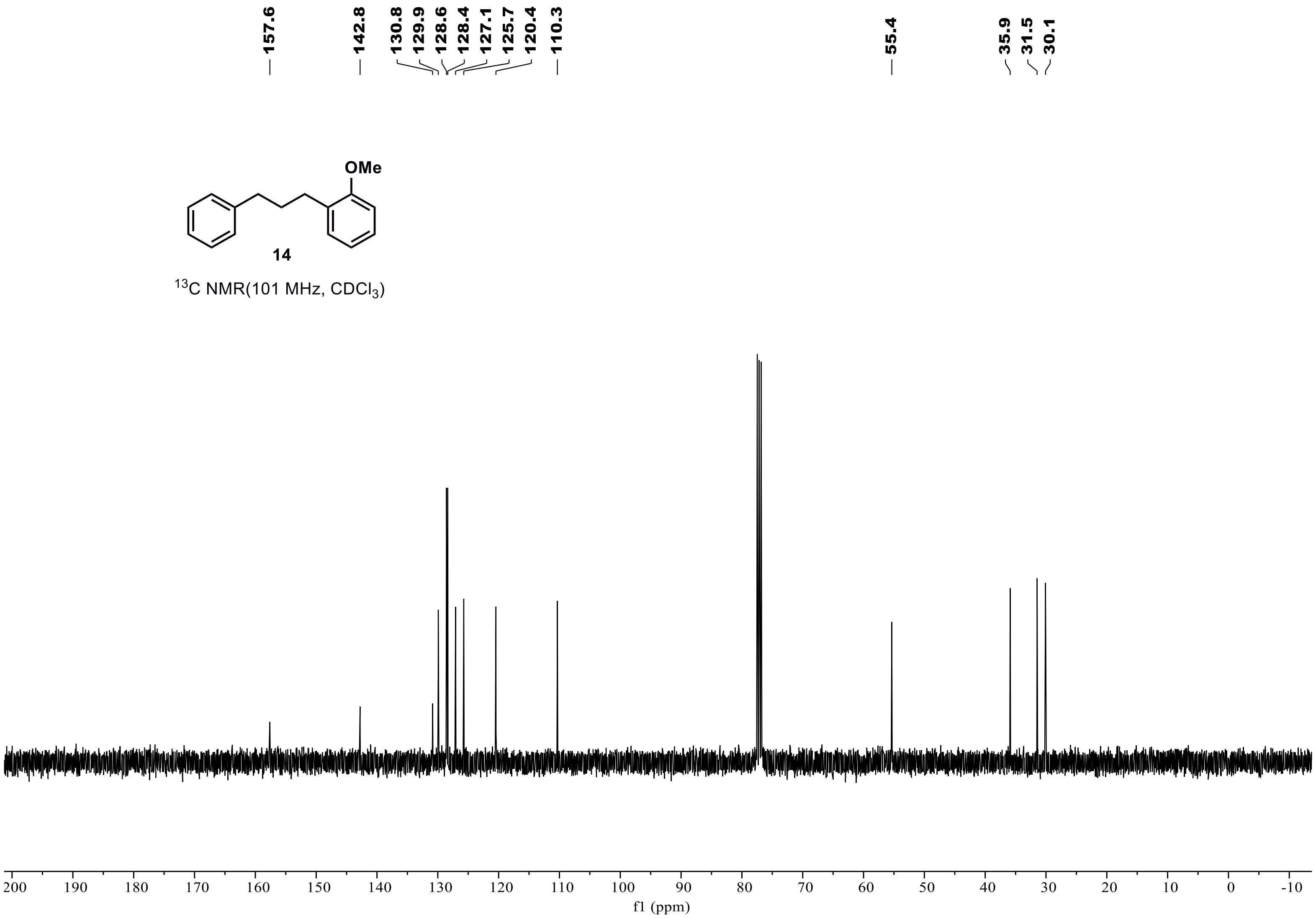




13

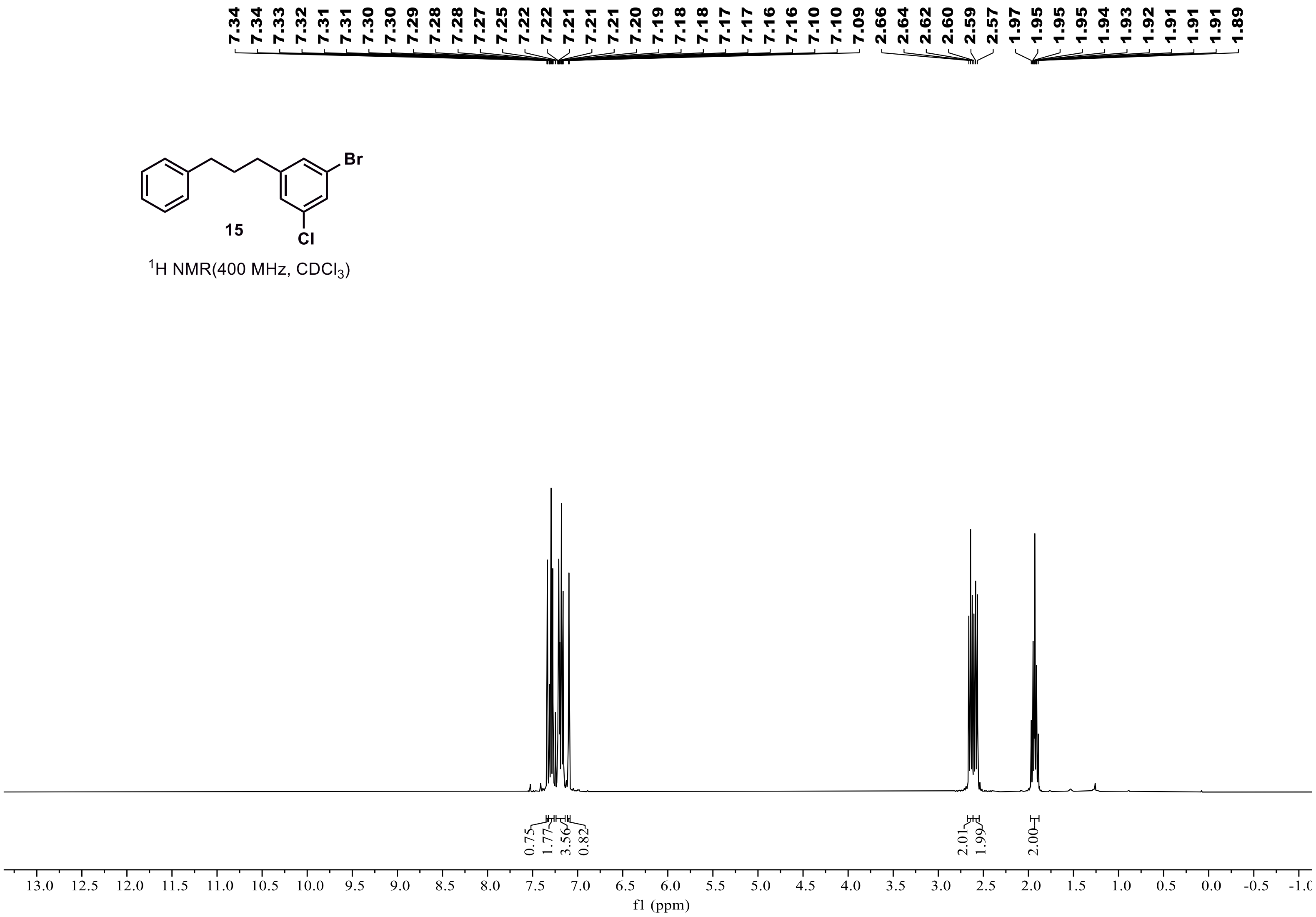
^{13}C NMR(101 MHz, $CDCl_3$)


7.30
7.30
7.29
7.28
7.28
7.28
7.28
7.27
7.26
7.26
7.26
7.24
7.22
7.21
7.21
7.20
7.20
7.19
7.19
7.19
7.18
7.17
7.16
7.16
7.14
7.14
7.13
7.12
6.90
6.90
6.88
6.88
6.87
6.86
6.86
6.85
6.84
6.83
3.81
2.69
2.67
2.67
2.67
2.65
1.97
1.95
1.95
1.95
1.94
1.93
1.93
1.92
1.91
1.91
1.89
OMe
14
^{1}H NMR(400 MHz, CDCl$_3$)
1.79
4.62
1.89
3.00
4.07
2.06
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)



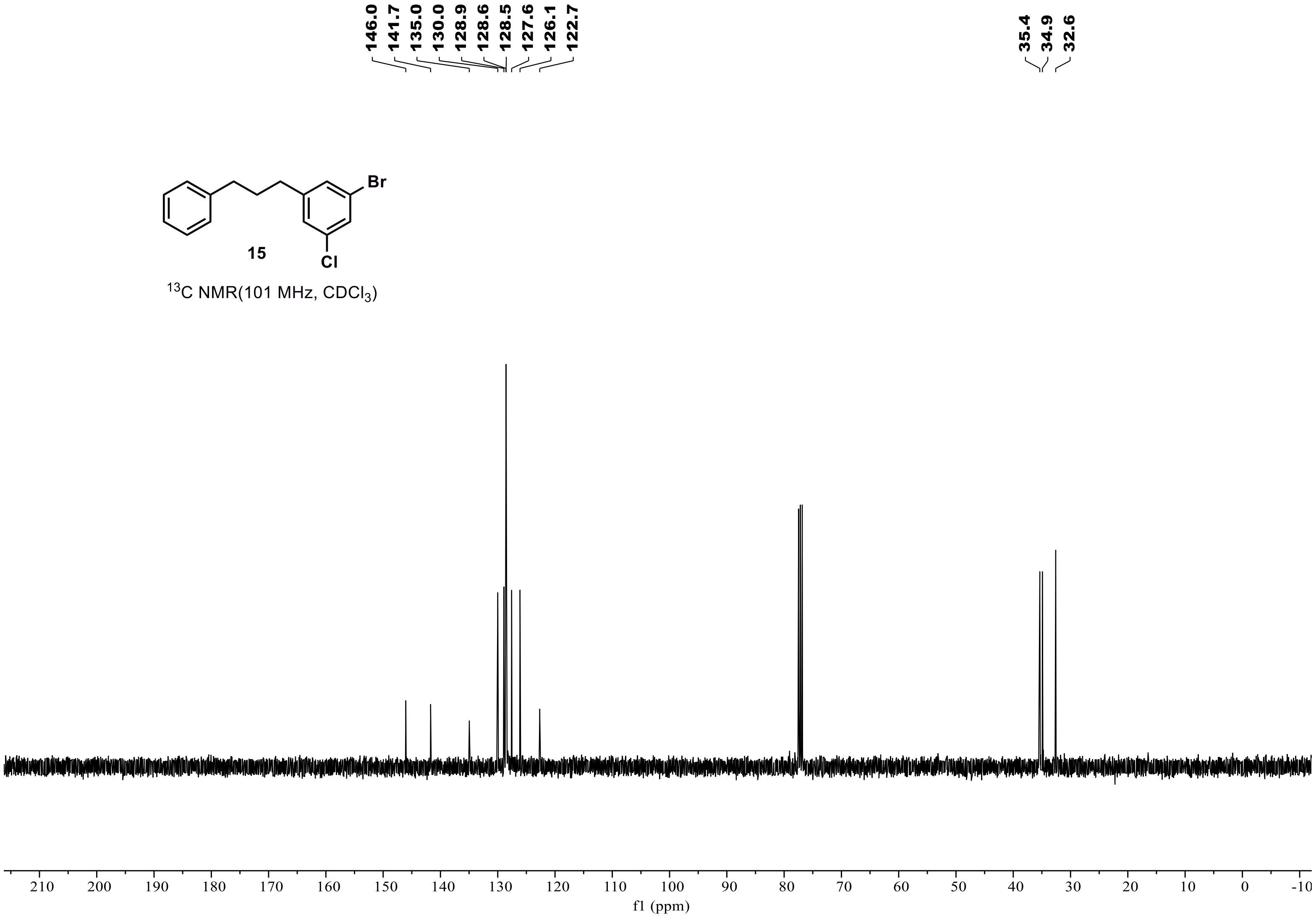
14

^{13}C NMR(101 MHz, CDCl$_3$)



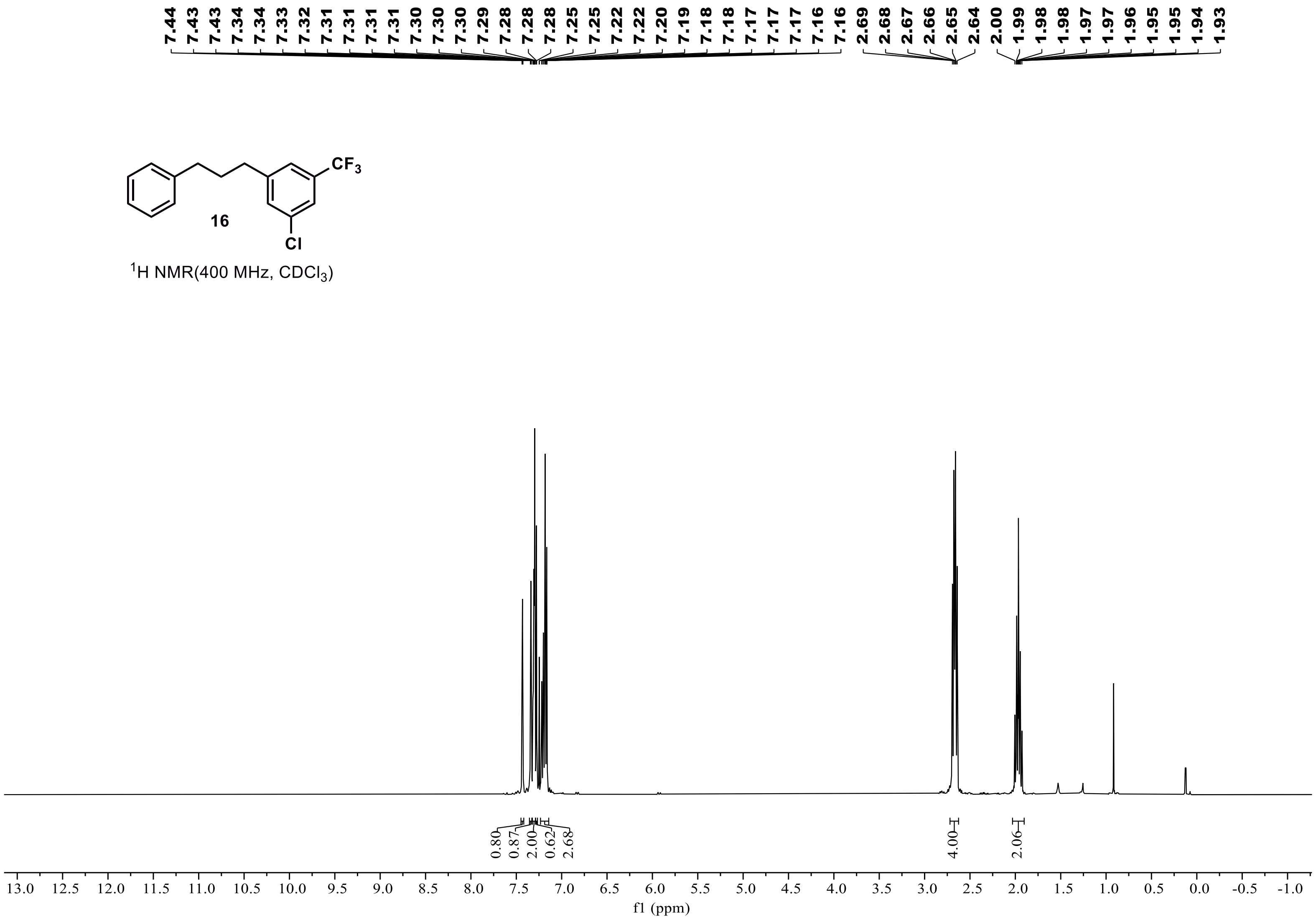
15

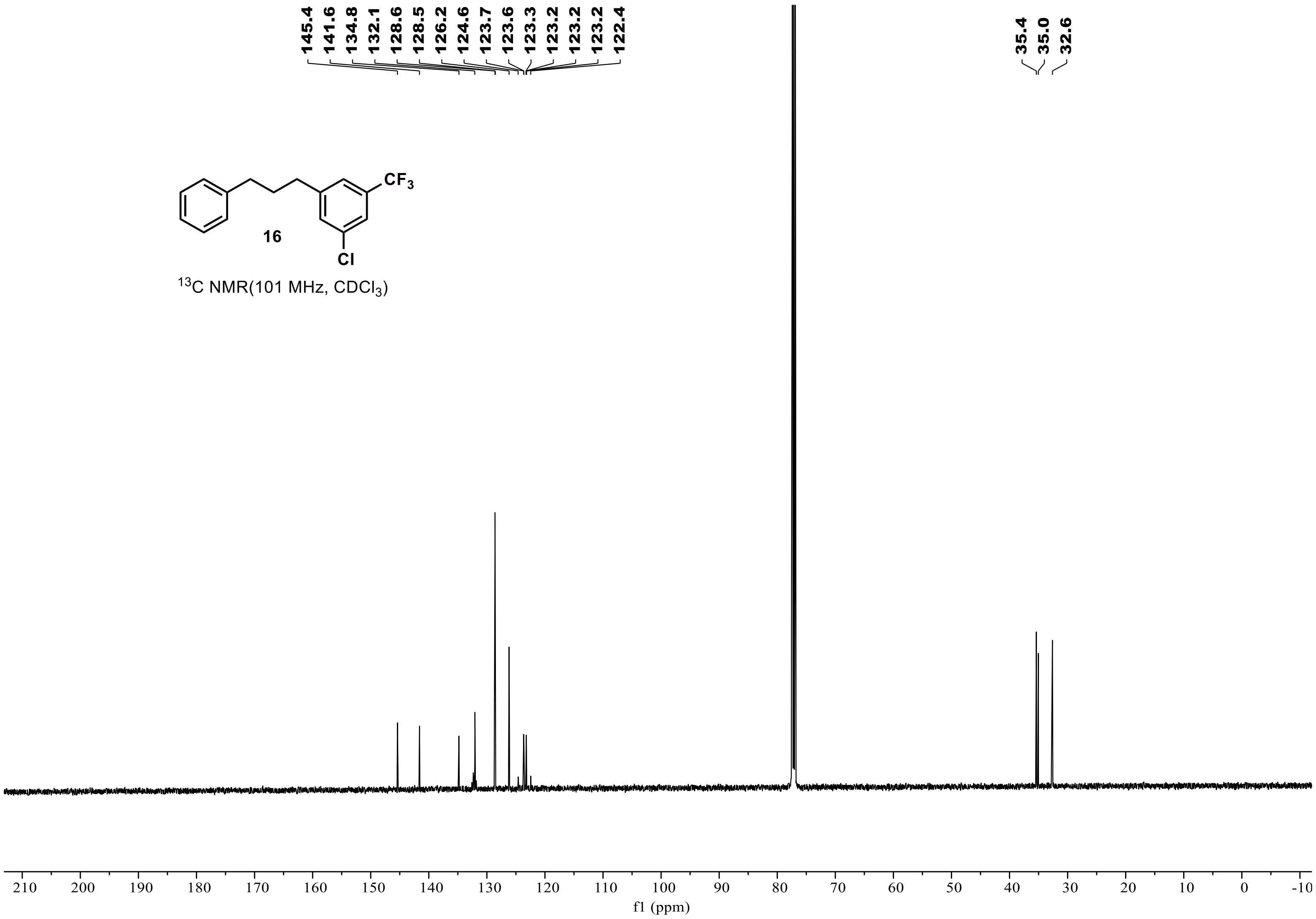
^{1}H NMR(400 MHz, $CDCl_3$)



15

^{13}C NMR(101 MHz, CDCl$_3$)

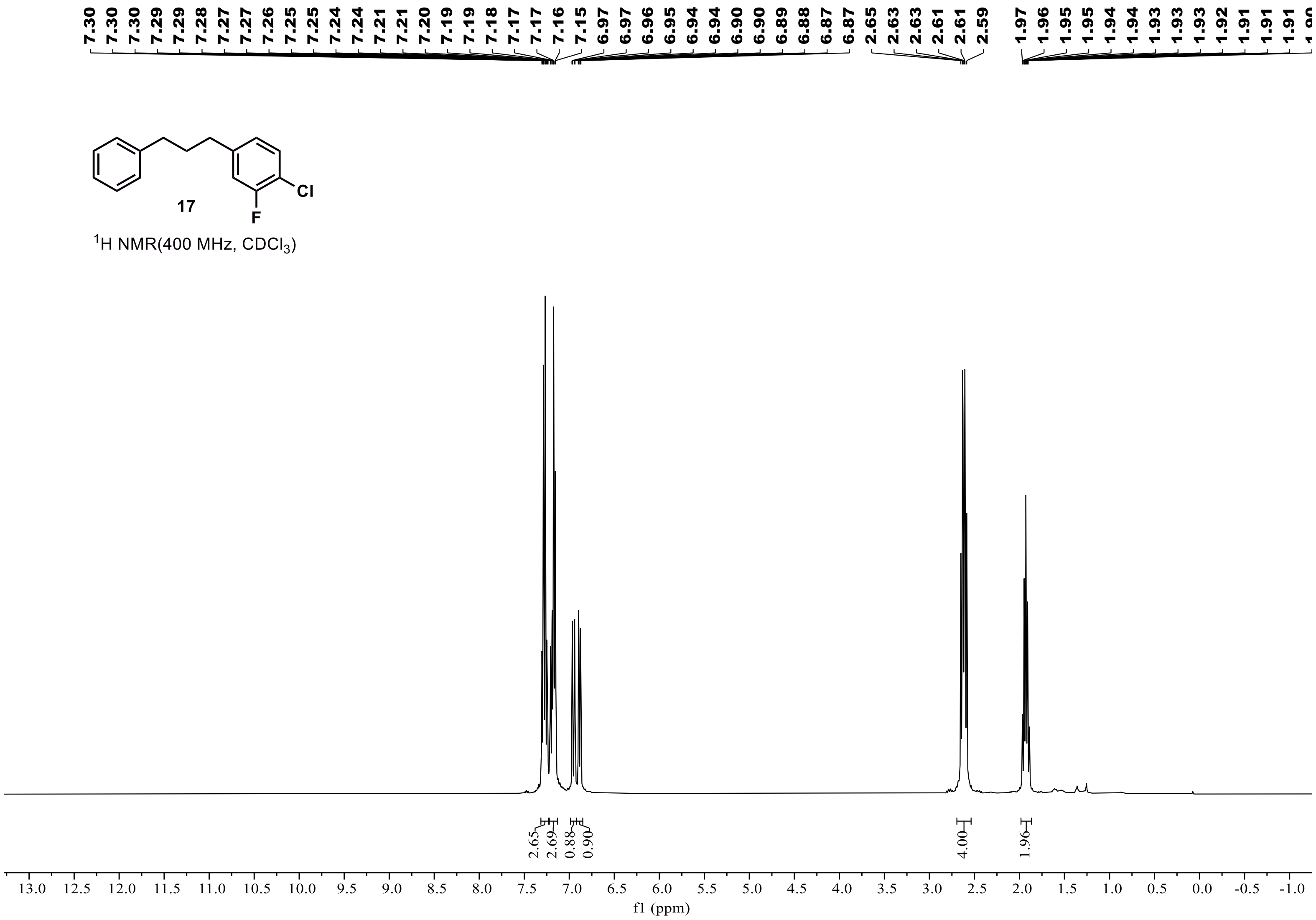
7.44
7.43
7.43
7.34
7.34
7.33
7.32
7.31
7.31
7.31
7.31
7.30
7.30
7.30
7.29
7.28
7.28
7.28
7.25
7.25
7.22
7.22
7.20
7.19
7.18
7.18
7.17
7.17
7.17
7.16
7.16
2.69
2.68
2.67
2.66
2.65
2.64
2.00
1.99
1.98
1.98
1.97
1.97
1.96
1.95
1.95
1.94
1.93
CF3
Cl
16
1H NMR(400 MHz, CDCl3)
0.80
0.87
2.00
0.62
2.68
4.00
2.06
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



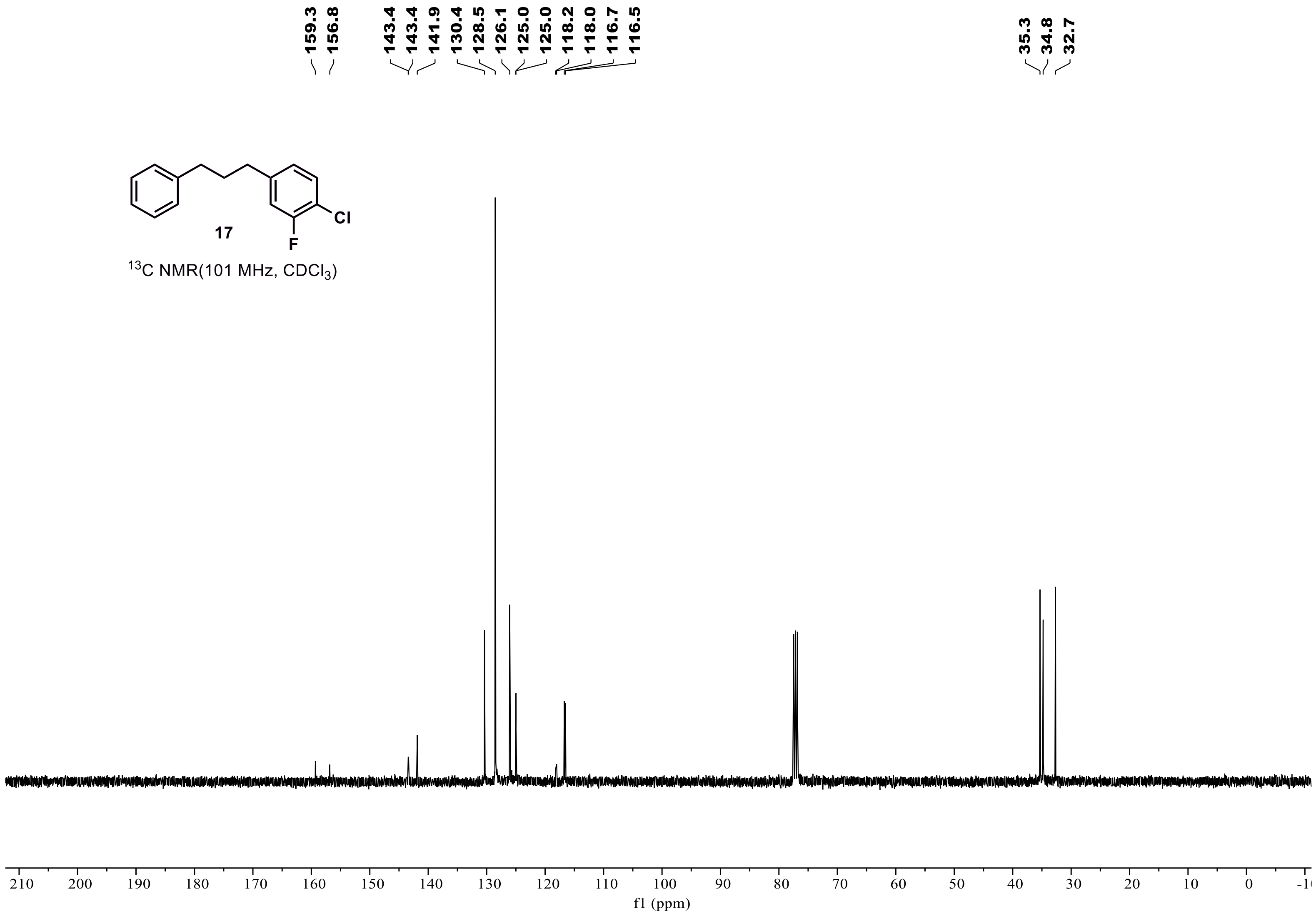
16

^{13}C NMR(101 MHz, CDCl$_3$)


-62.77
CF3
16
Cl
19F NMR(376 MHz, CDCl3)
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

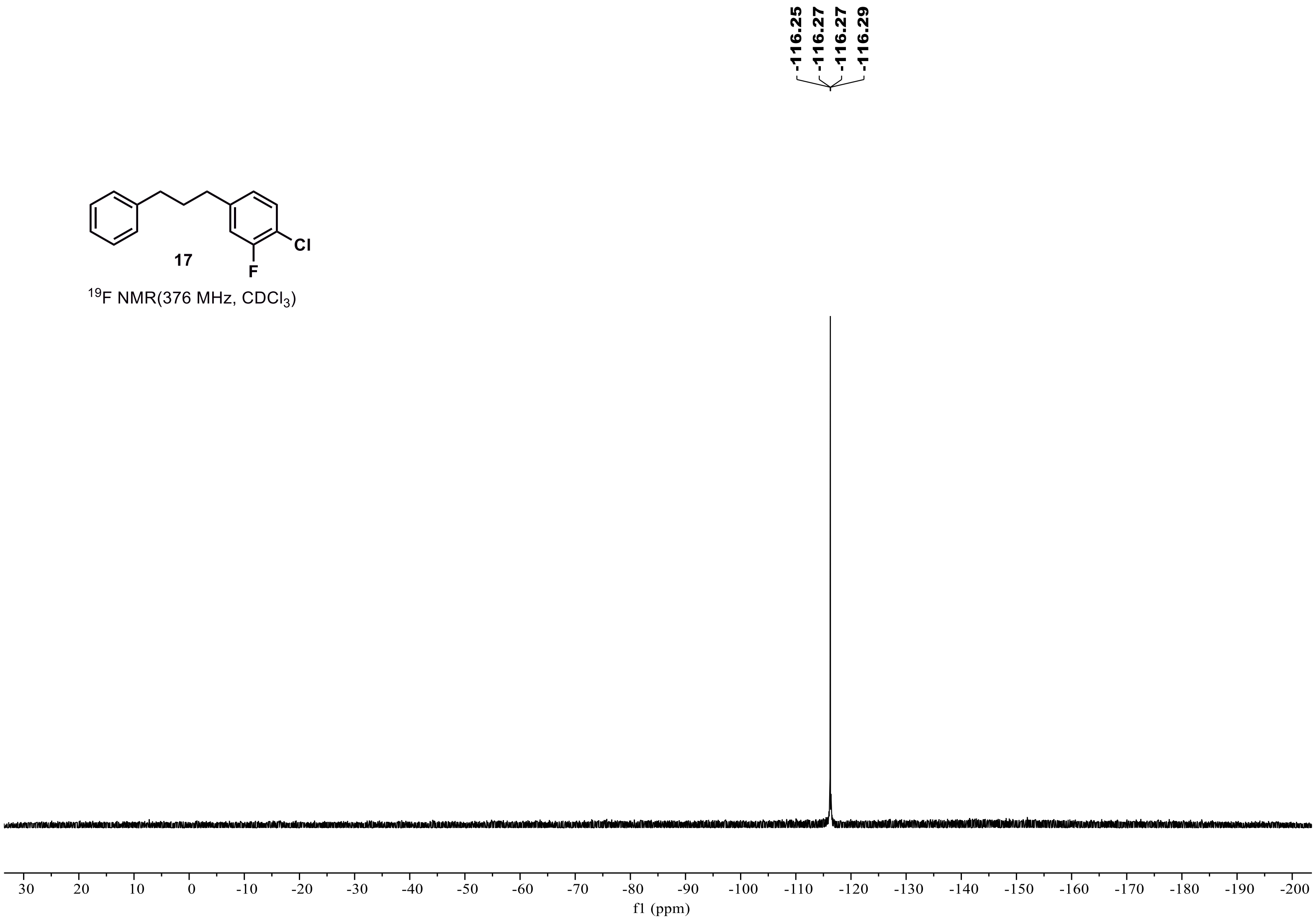


^{1}H NMR(400 MHz, CDCl$_3$)



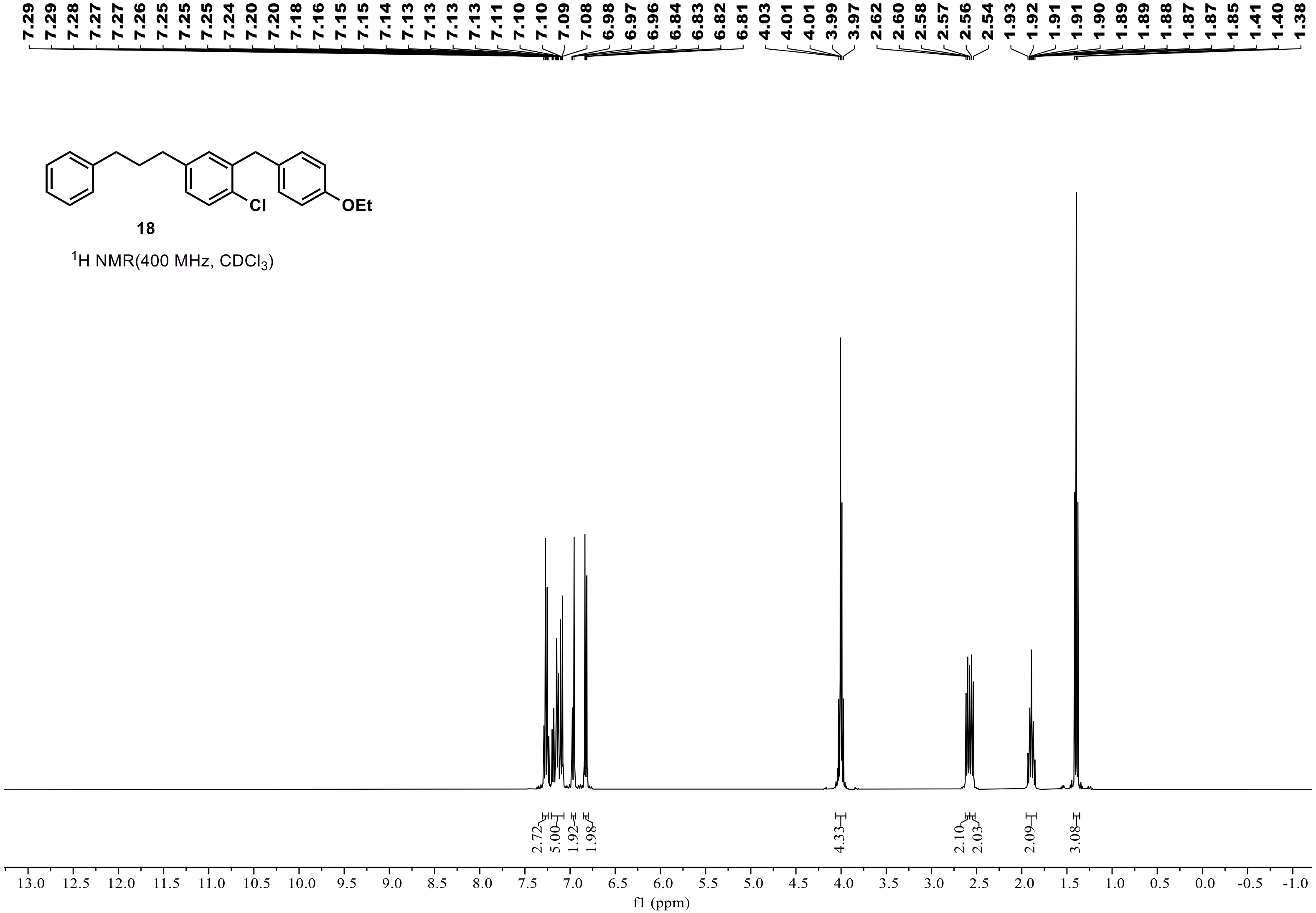
17

^{13}C NMR(101 MHz, $CDCl_3$)



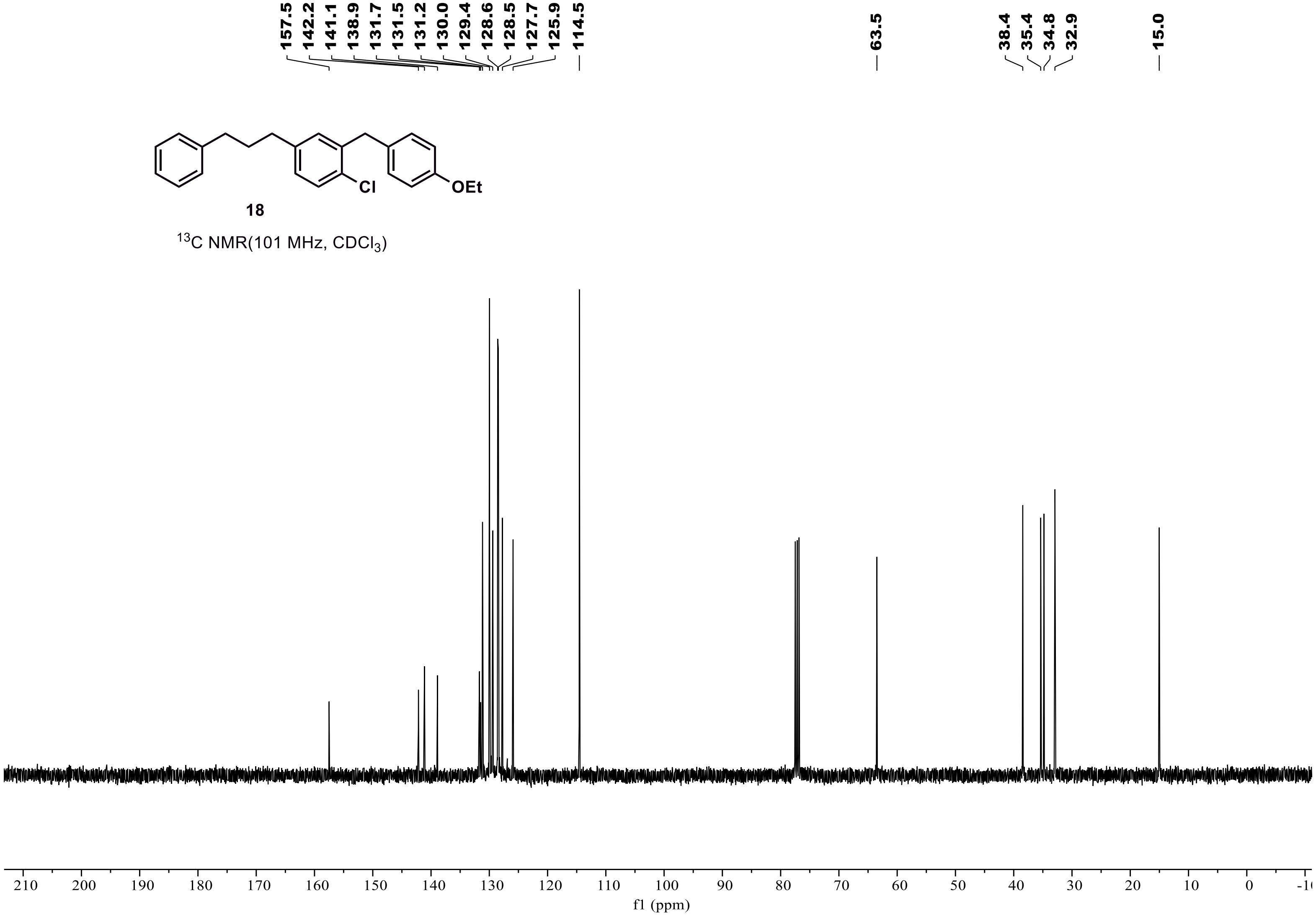
17

^{19}F NMR(376 MHz, $CDCl_3$)



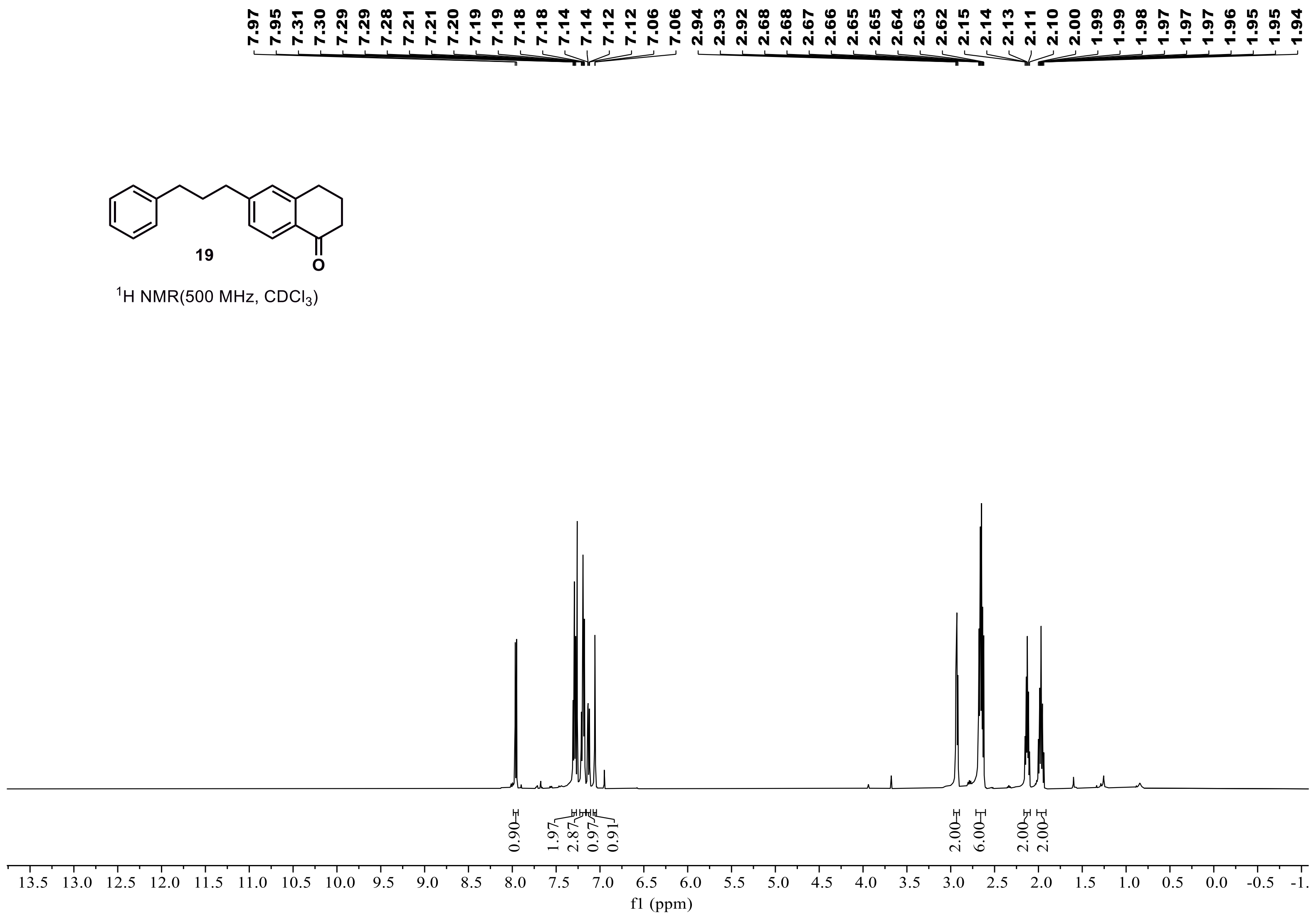
18

^{1}H NMR(400 MHz, $CDCl_3$)



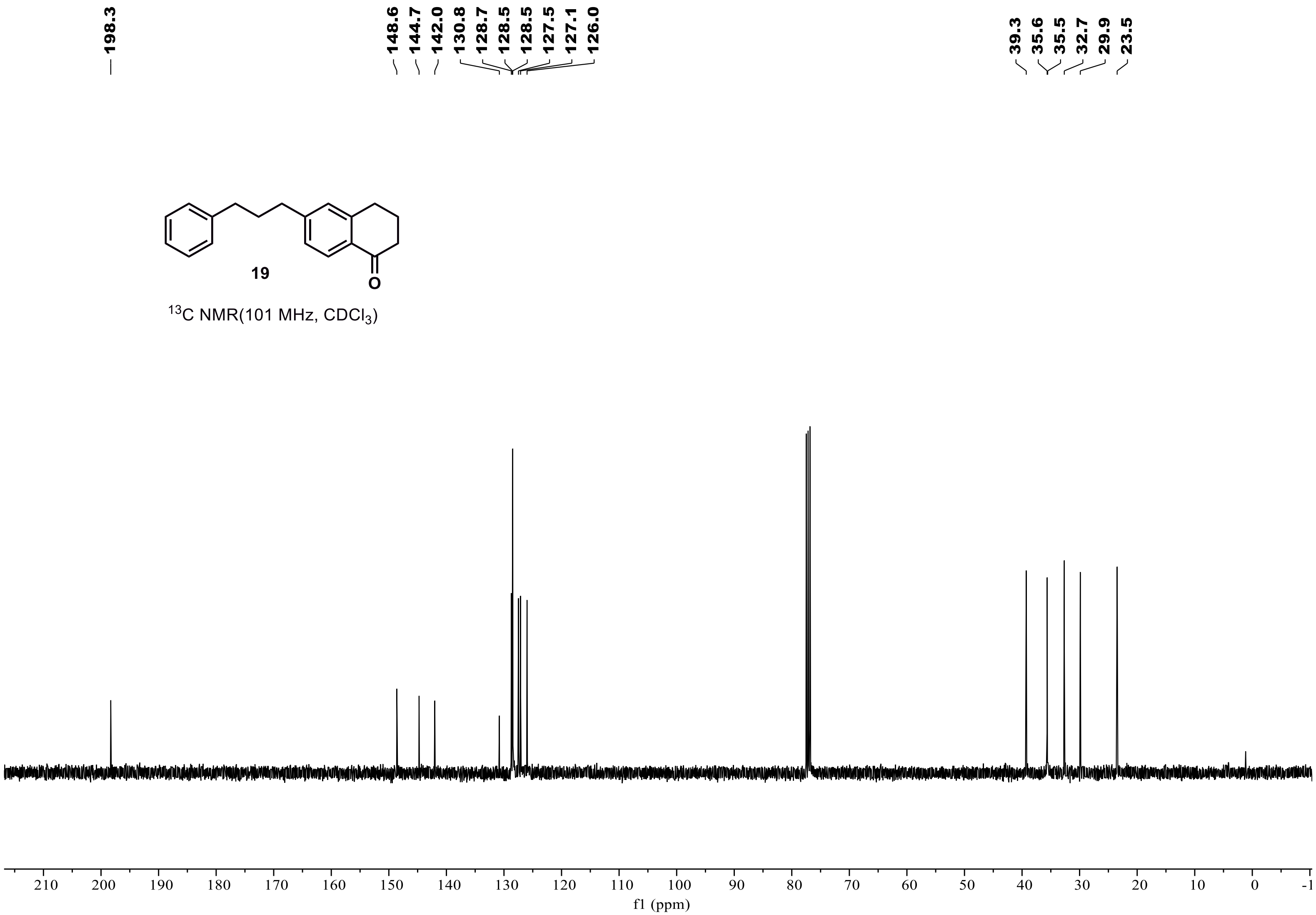
18

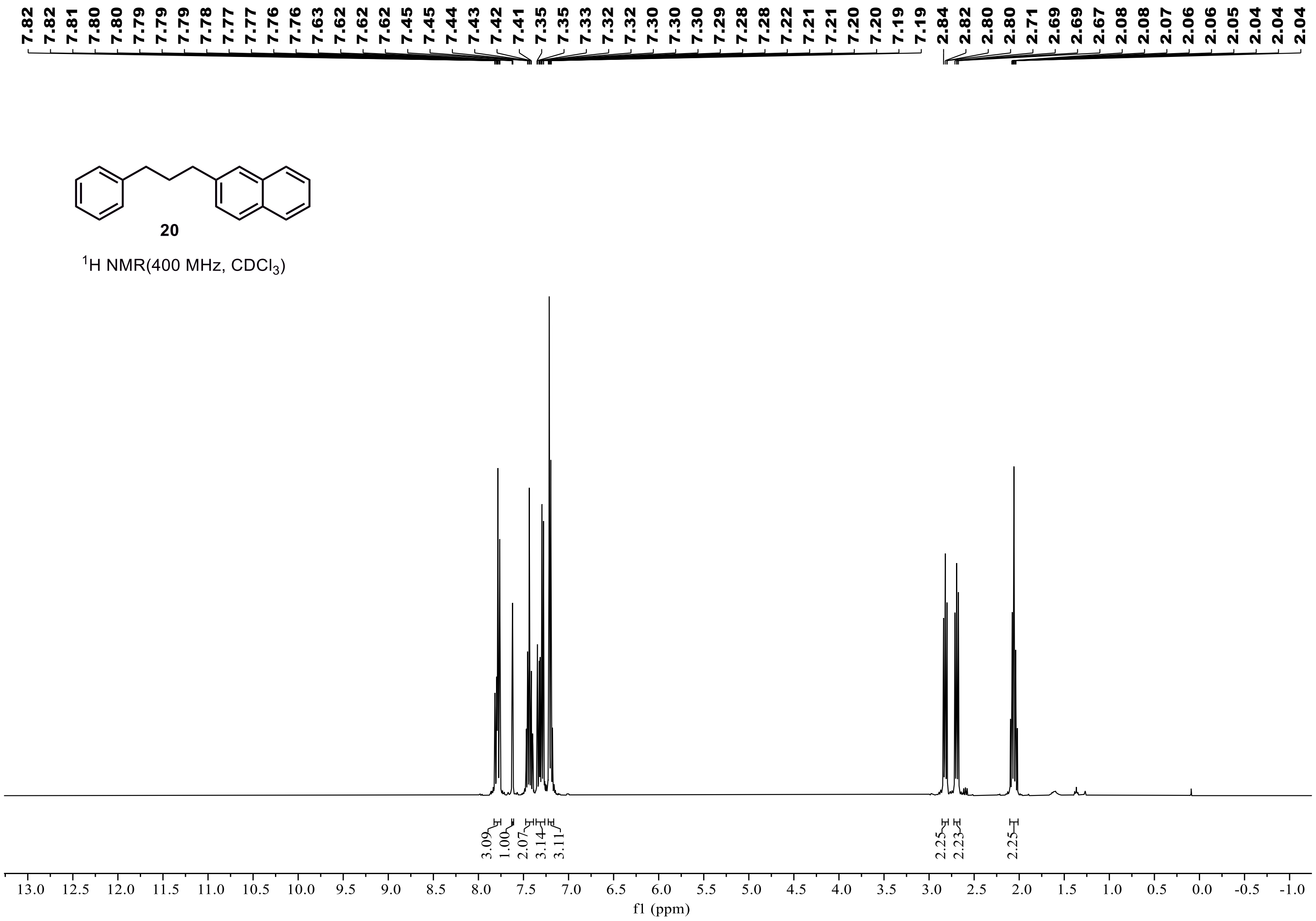
^{13}C NMR(101 MHz, CDCl$_3$)

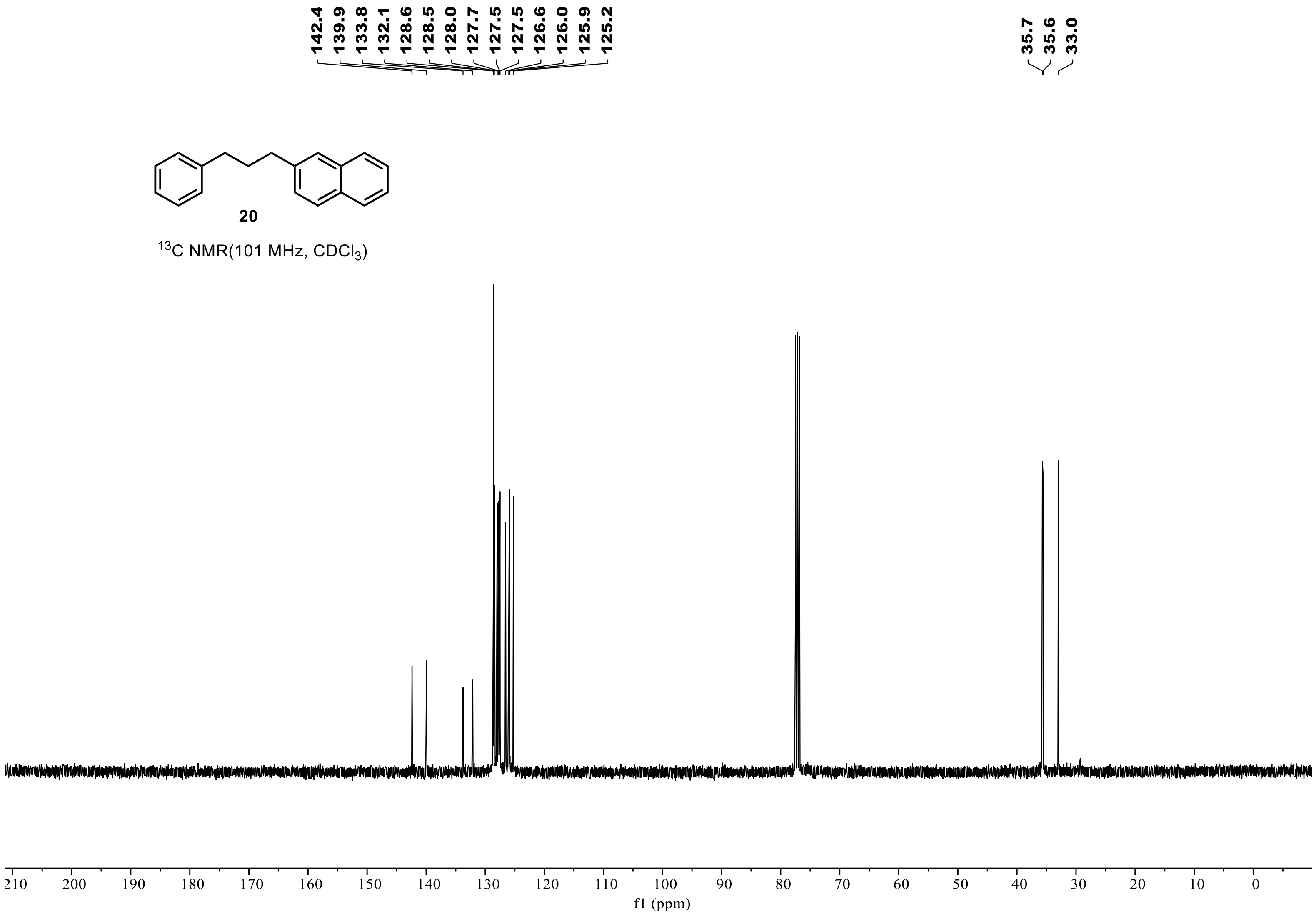


19

^{1}H NMR(500 MHz, CDCl$_3$)

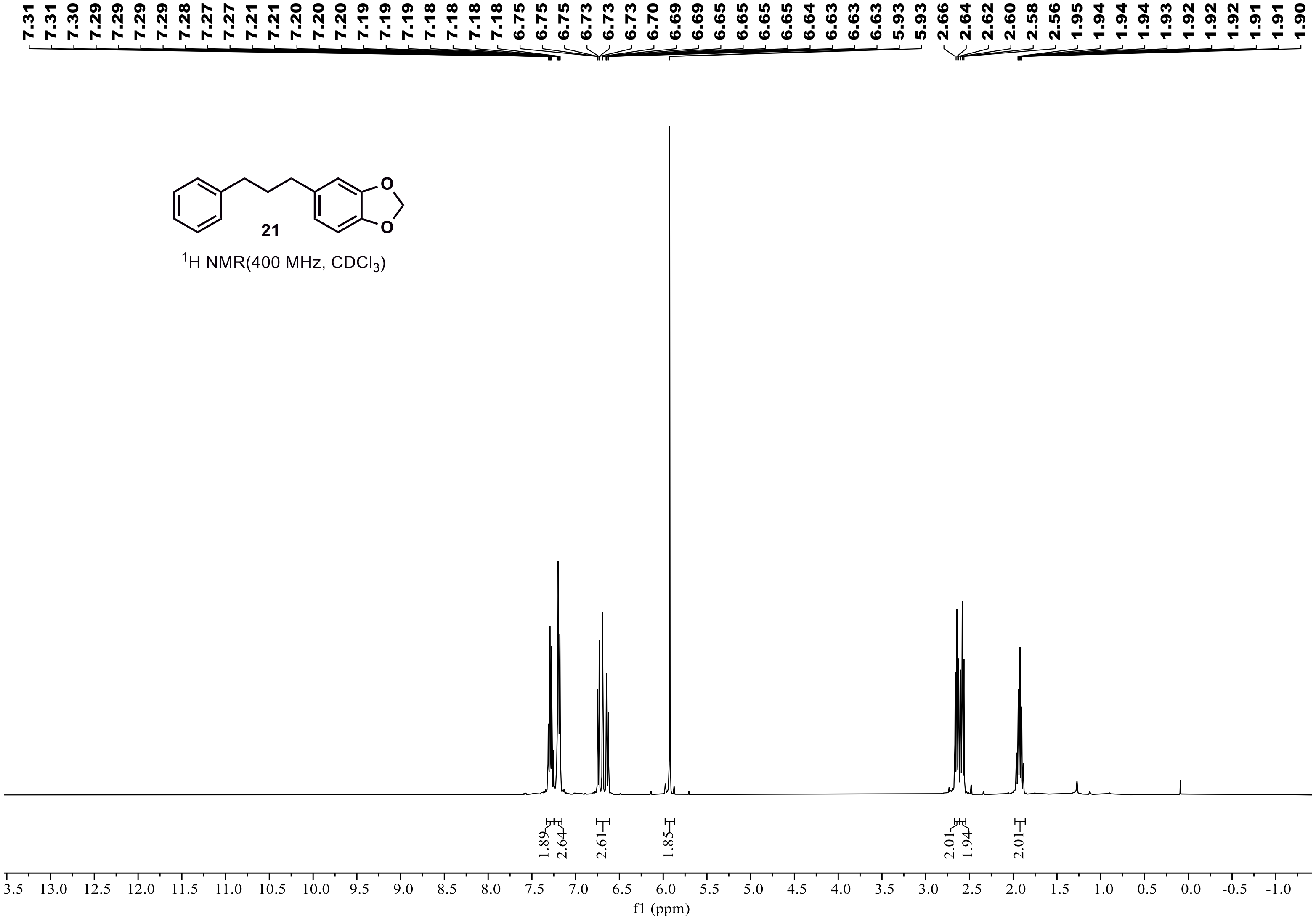

198.3
148.6
144.7
142.0
130.8
128.7
128.5
128.5
127.5
127.1
126.0
39.3
35.6
35.5
32.7
29.9
23.5
19
O
^{13}C NMR(101 MHz, CDCl$_3$)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)


7.82 7.82 7.81 7.80 7.80 7.79 7.79 7.79 7.78 7.77 7.77 7.76 7.76 7.63 7.62 7.62 7.62 7.45 7.45 7.44 7.43 7.42 7.41 7.35 7.35 7.33 7.32 7.32 7.30 7.30 7.30 7.29 7.28 7.28 7.22 7.21 7.21 7.20 7.20 7.19 2.84 2.82 2.80 2.80 2.71 2.69 2.69 2.67 2.08 2.08 2.07 2.06 2.06 2.05 2.04 2.04 2.04
20
^{1}H NMR(400 MHz, CDCl$_3$)
3.09
1.00
2.07
3.14
3.11
2.25
2.23
2.25
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

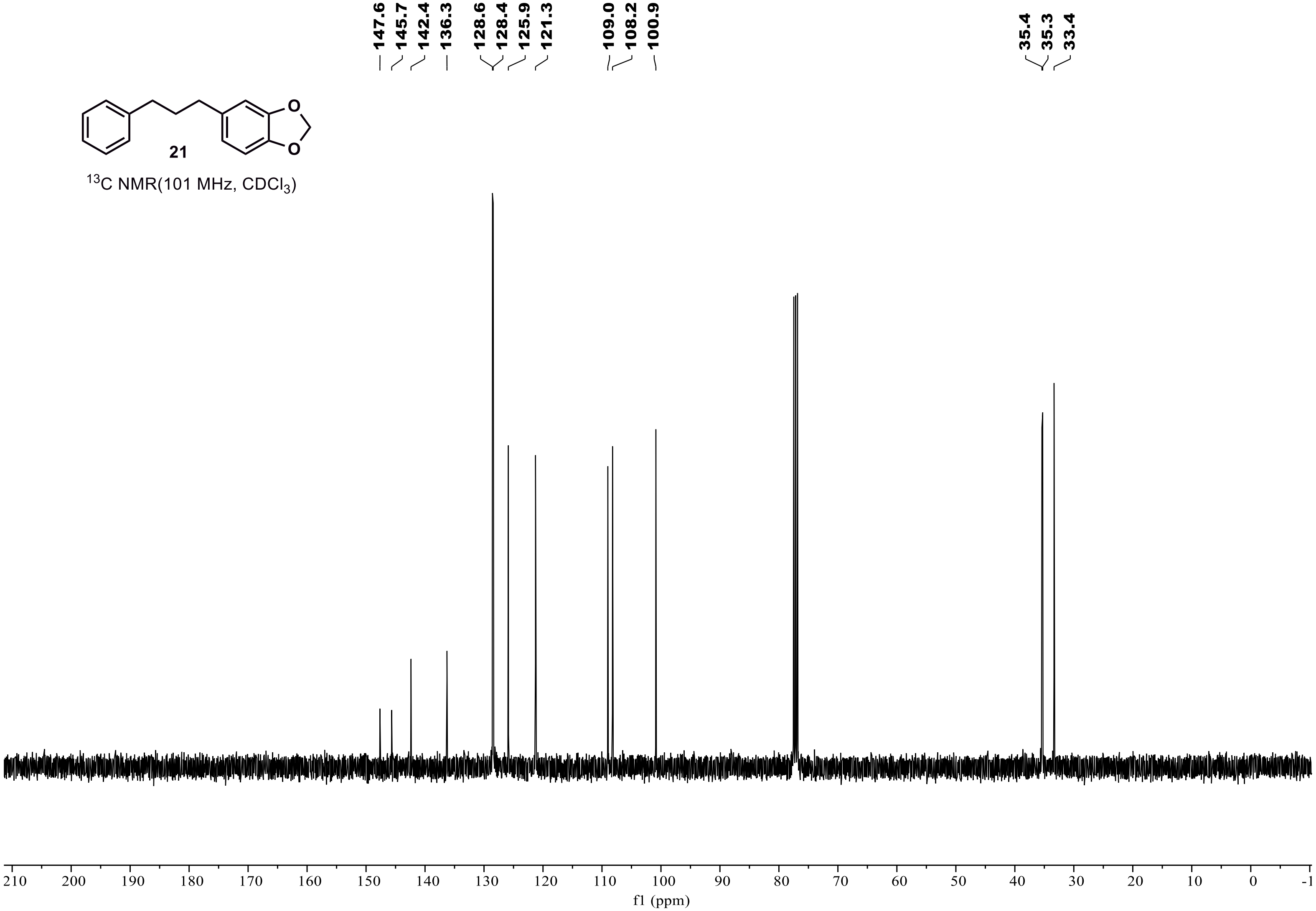


20

^{13}C NMR(101 MHz, CDCl$_3$)

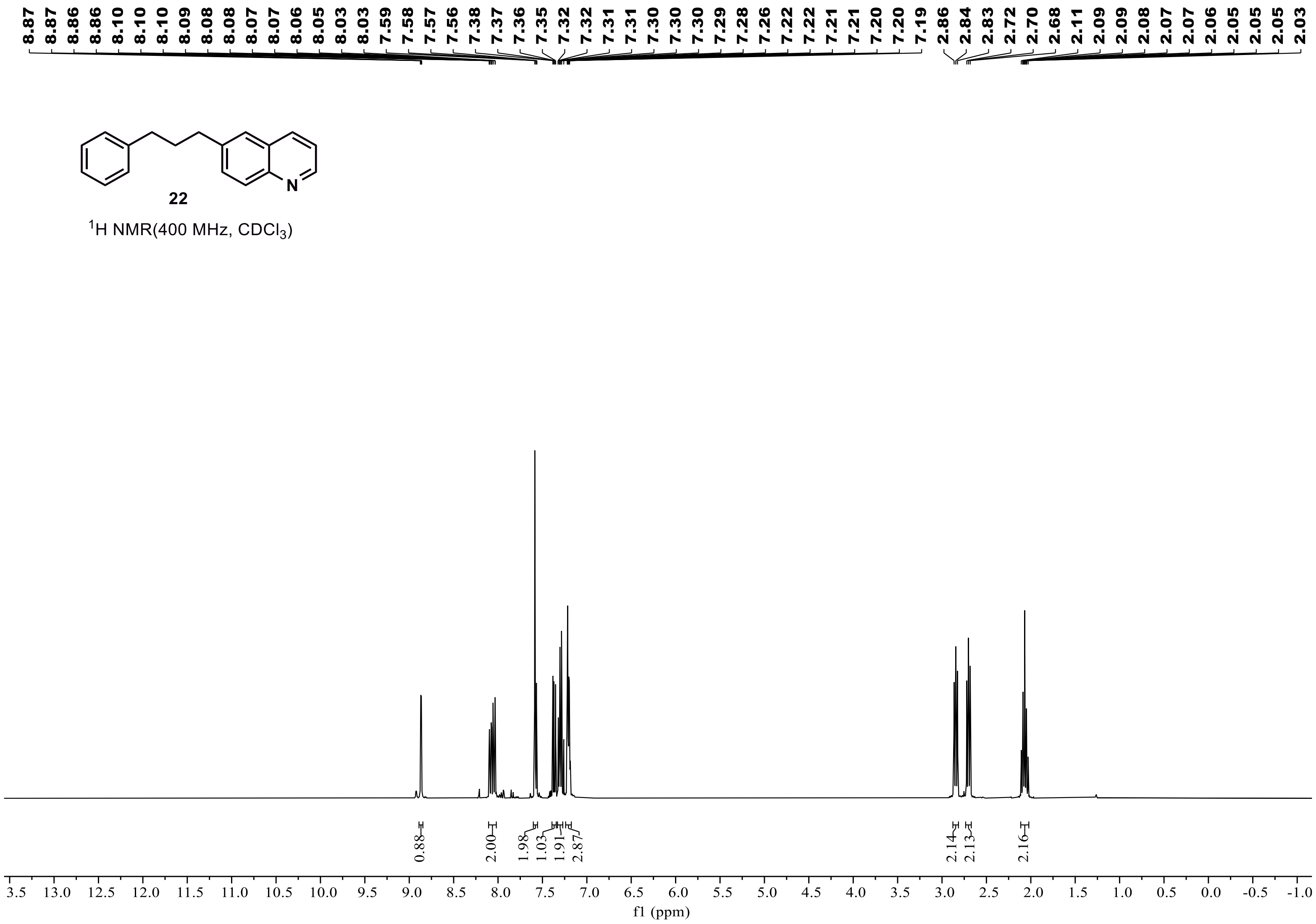

21

^{1}H NMR(400 MHz, CDCl$_3$)



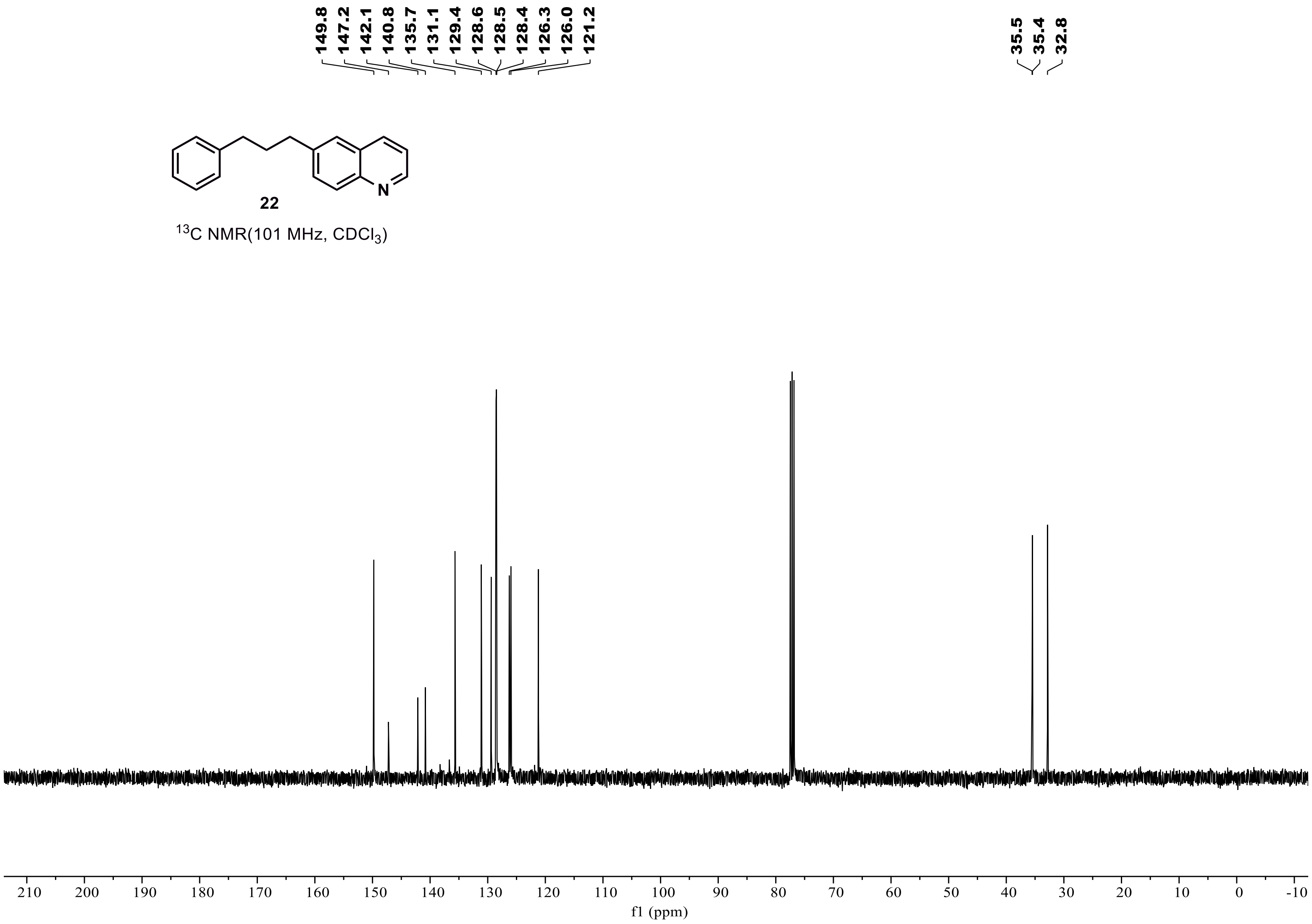
21

^{13}C NMR(101 MHz, CDCl$_3$)



22

^{1}H NMR(400 MHz, $CDCl_3$)

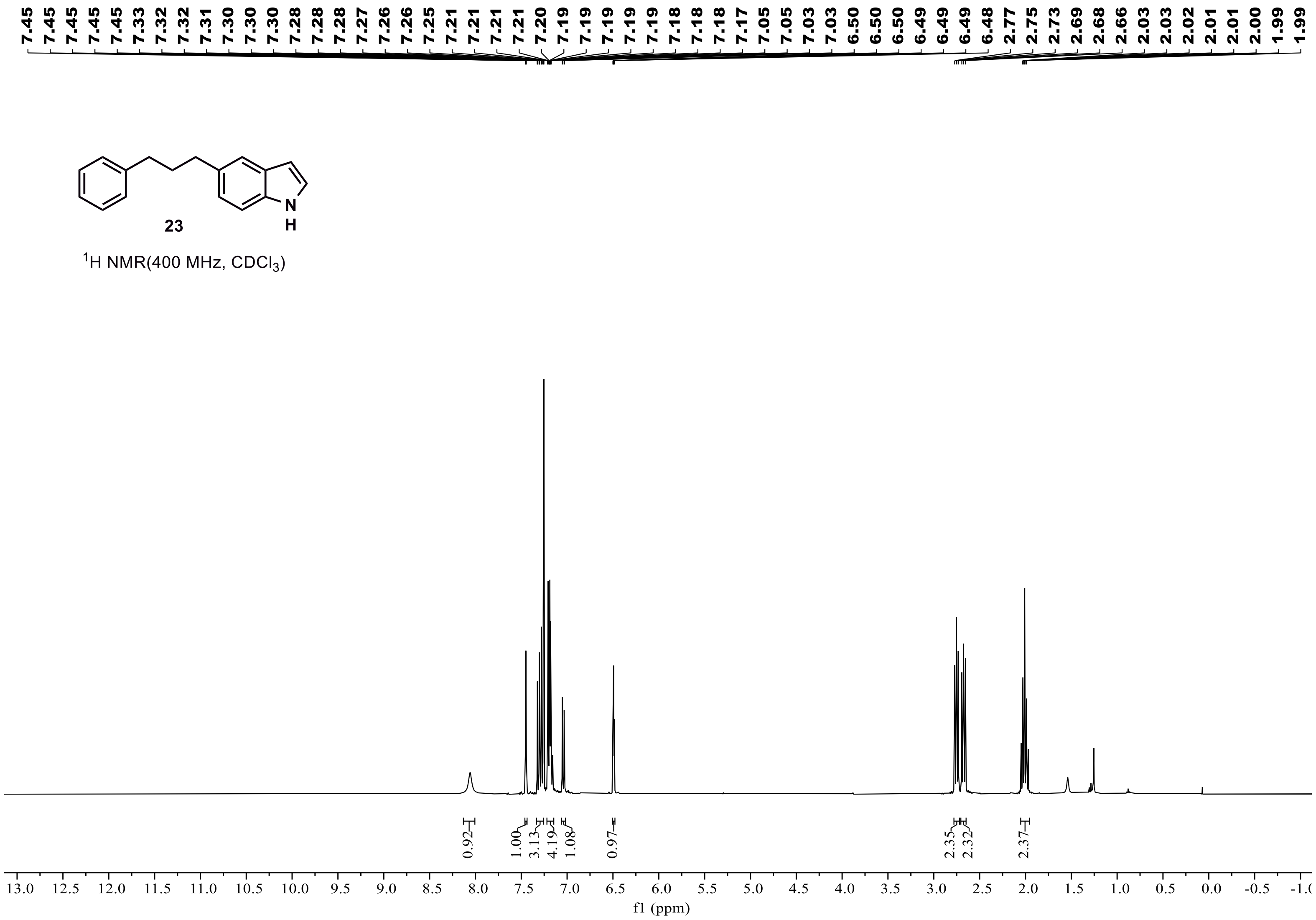


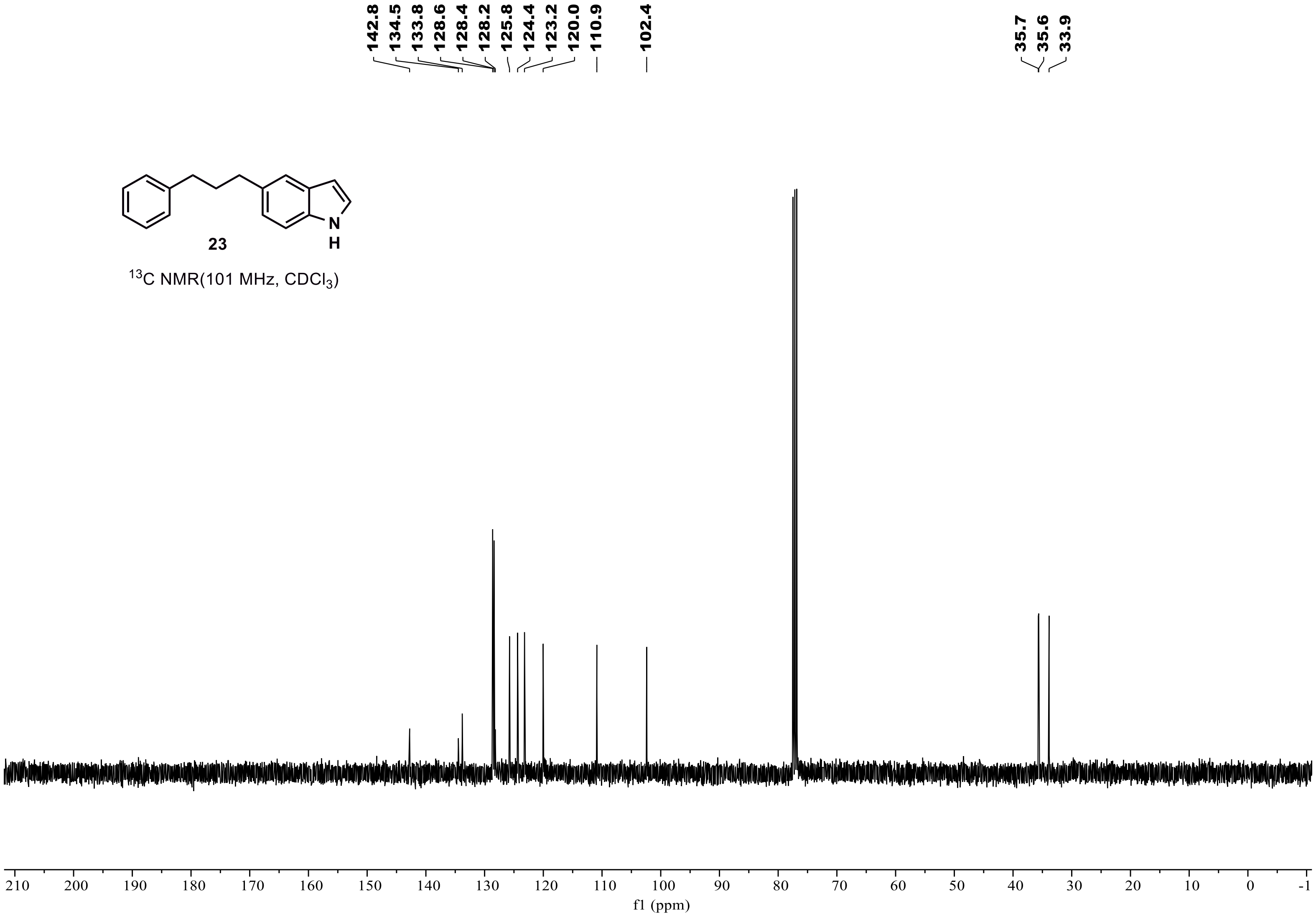
22

^{13}C NMR(101 MHz, CDCl$_3$)

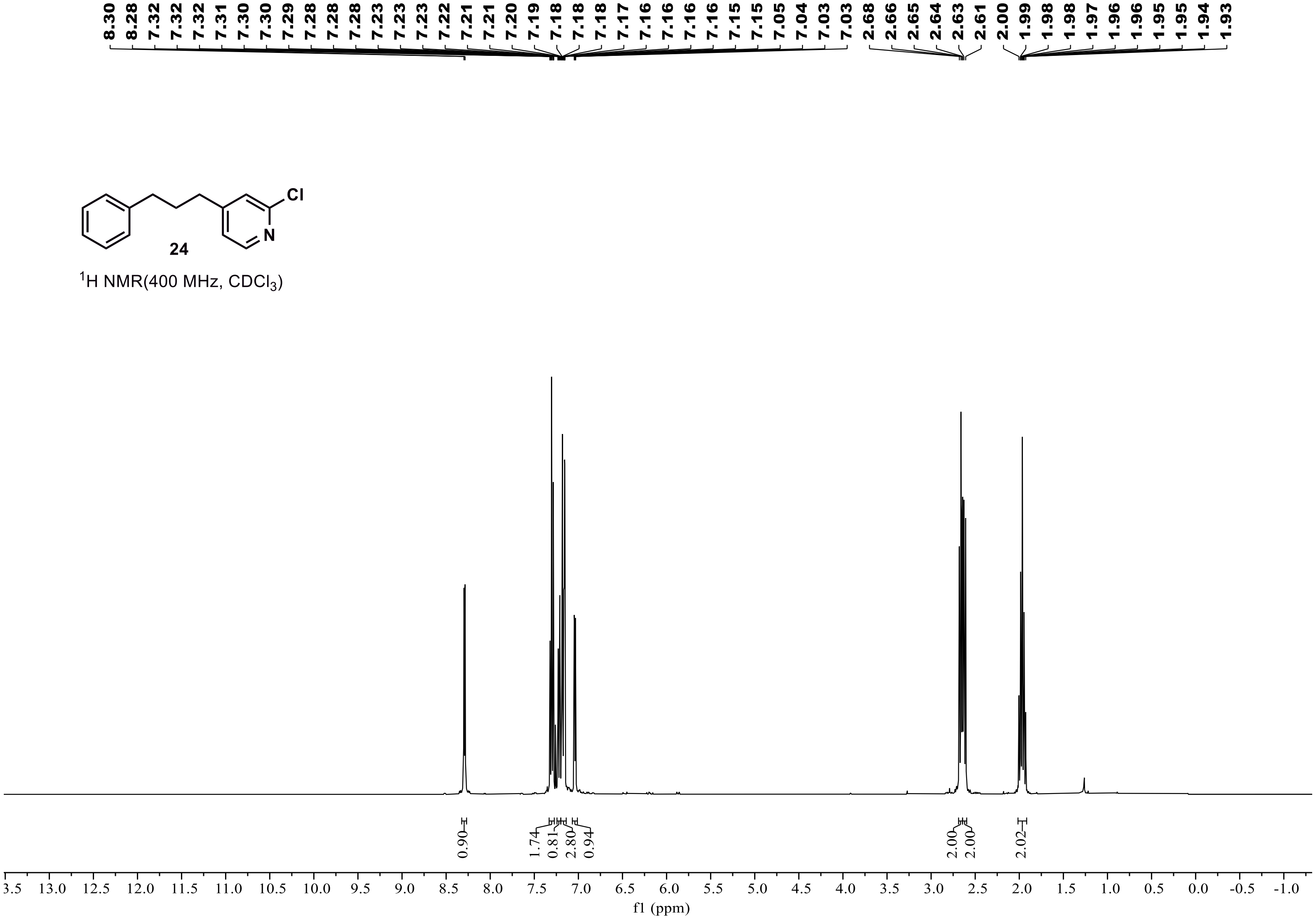
23

^1H NMR(400 MHz, $CDCl_3$)



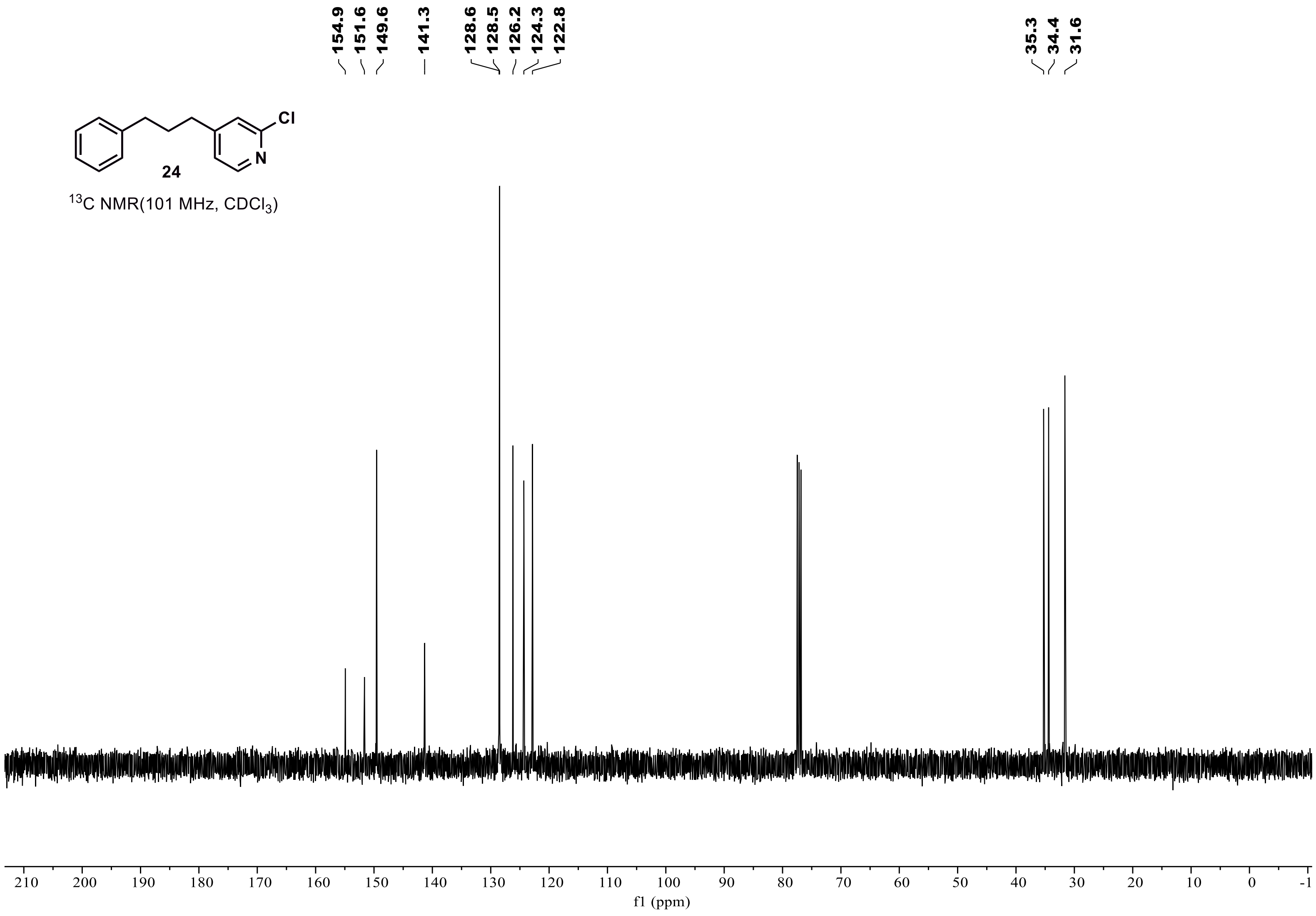

23

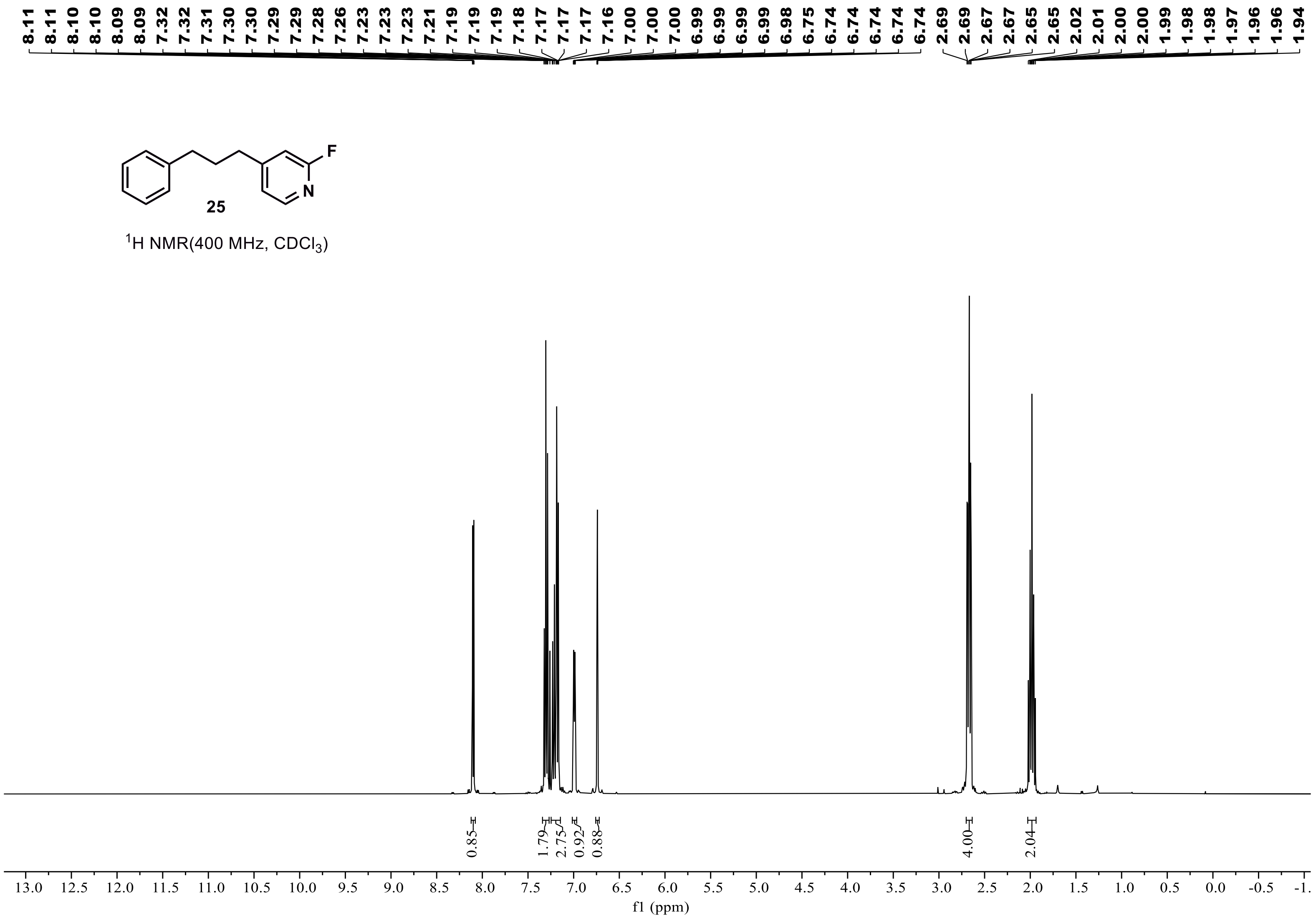
^{13}C NMR(101 MHz, $CDCl_3$)

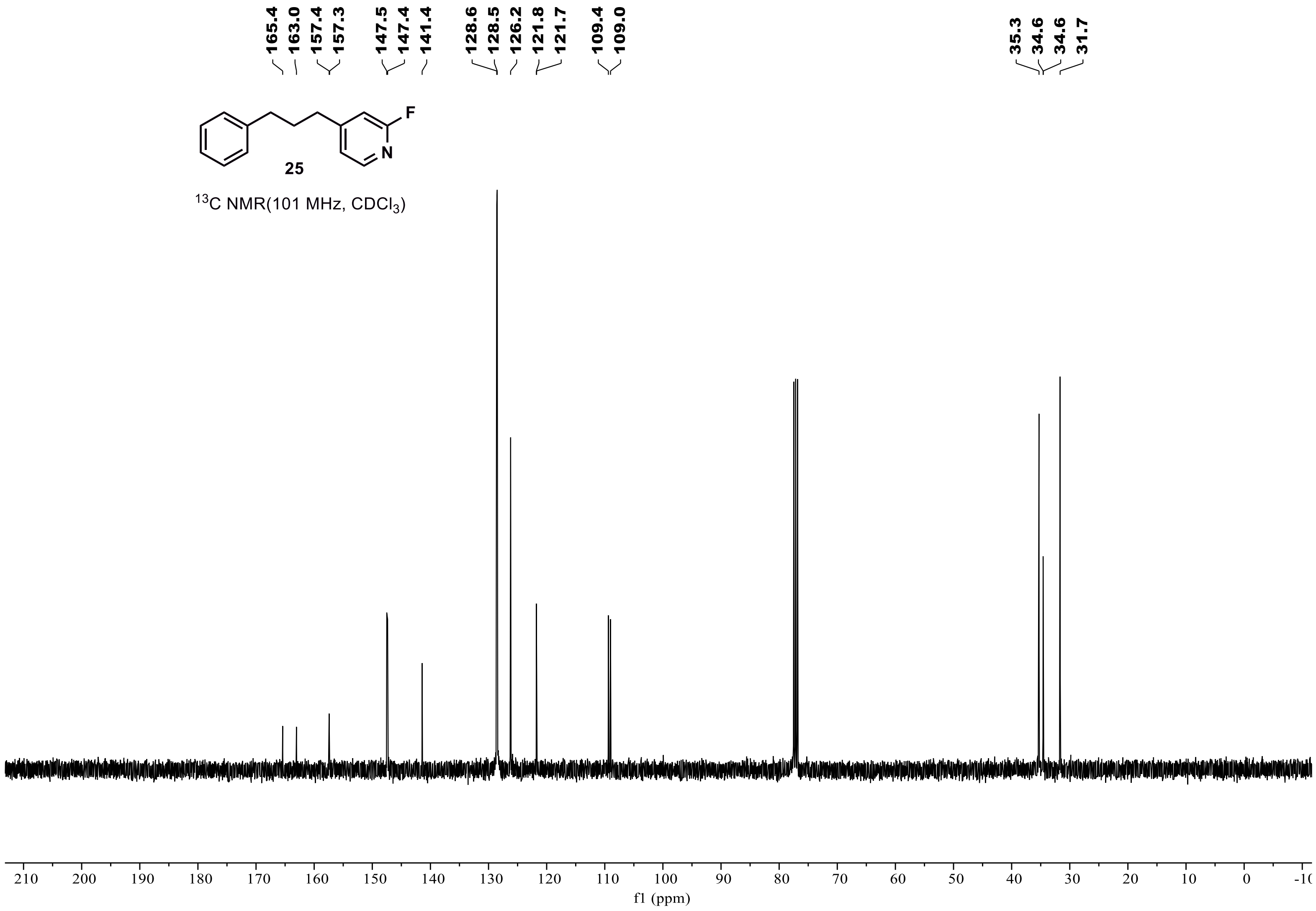


24

^{1}H NMR(400 MHz, CDCl$_3$)

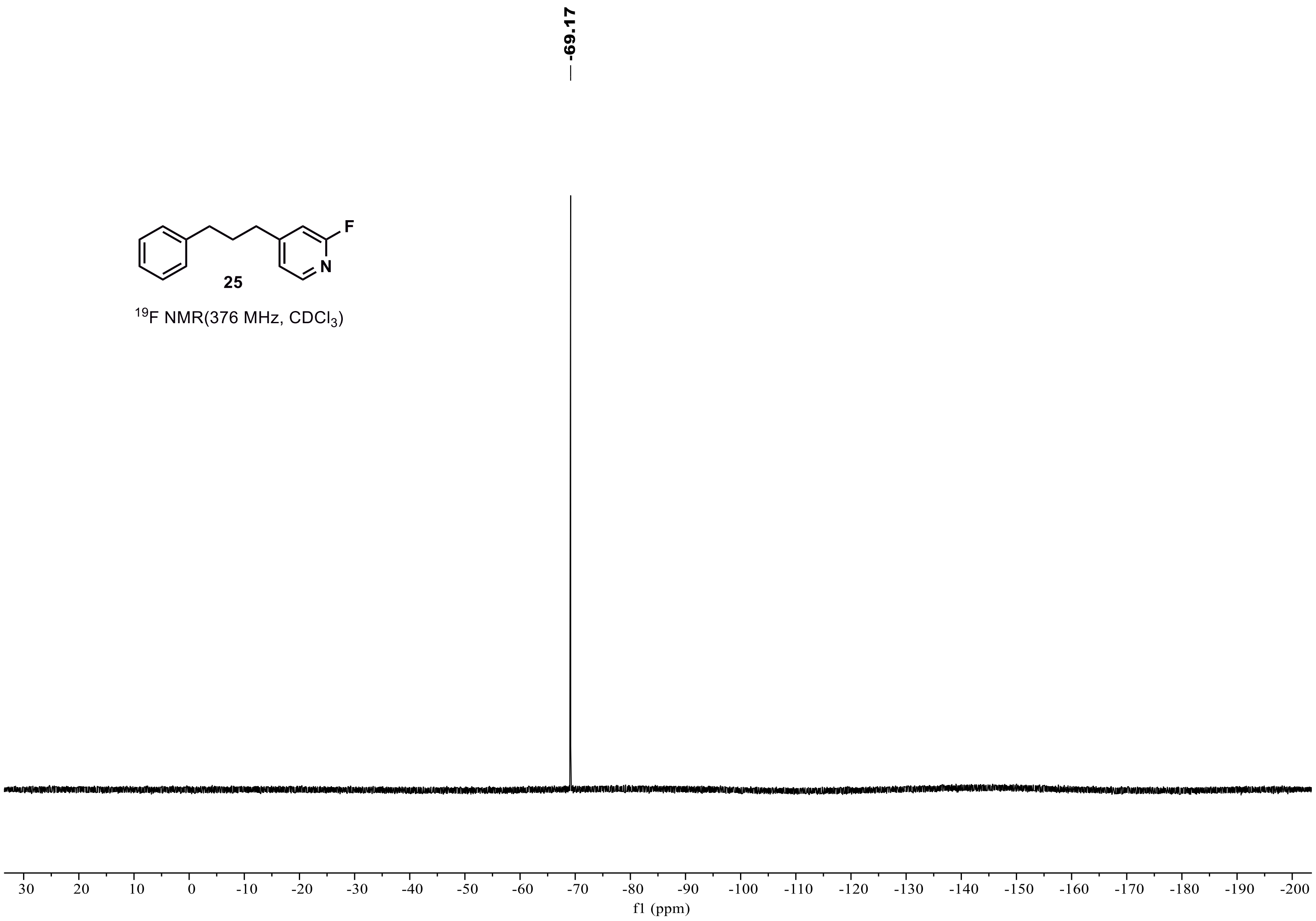

154.9
151.6
149.6
141.3
128.6
128.5
126.2
124.3
122.8
35.3
34.4
31.6
Cl
N
24
^{13}C NMR(101 MHz, CDCl$_3$)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

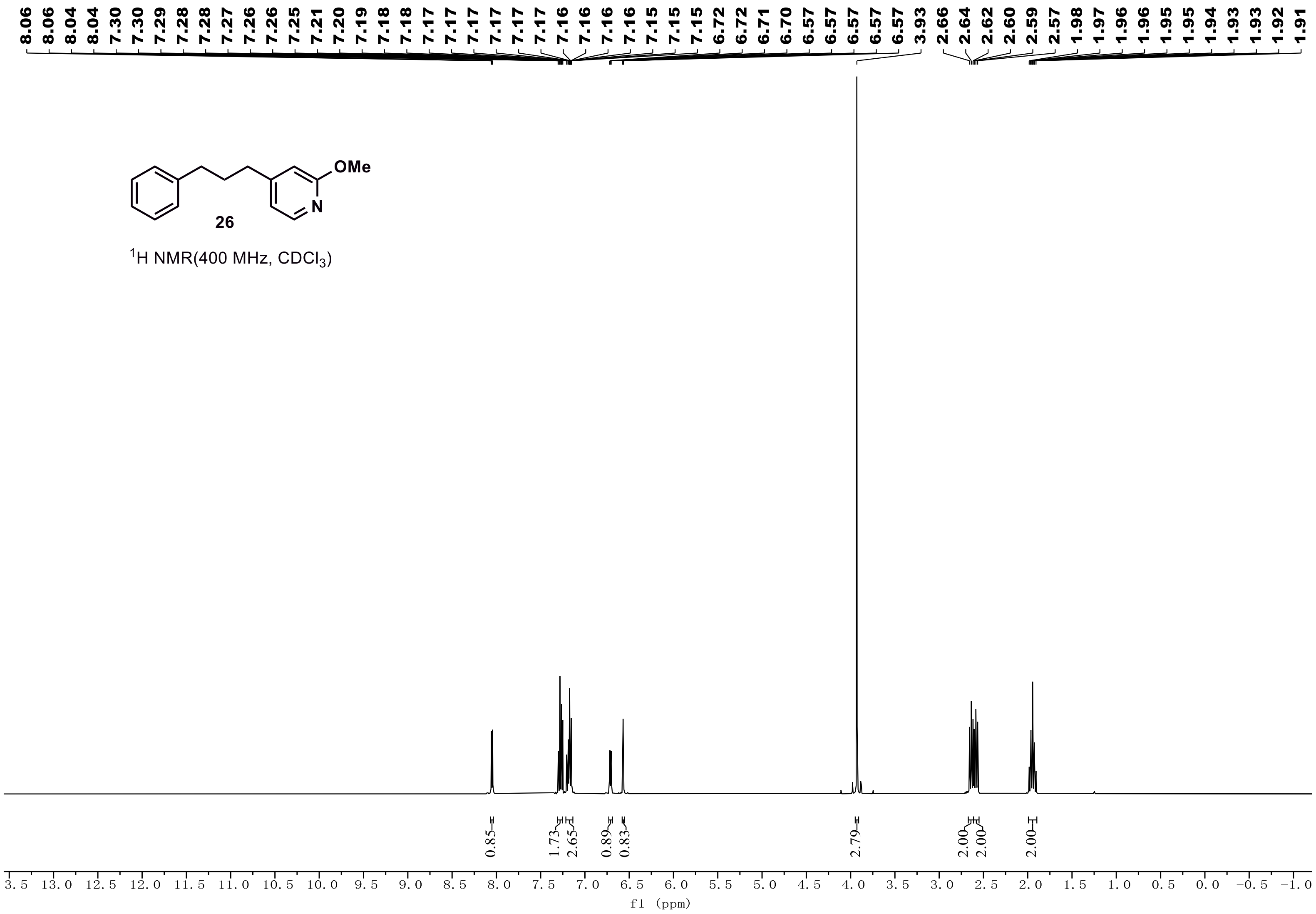
8.11
8.11
8.10
8.10
8.09
8.09
7.32
7.32
7.31
7.30
7.30
7.29
7.29
7.28
7.26
7.23
7.23
7.23
7.21
7.19
7.19
7.19
7.18
7.17
7.17
7.17
7.16
7.00
7.00
7.00
6.99
6.99
6.99
6.98
6.75
6.74
6.74
6.74
6.74
6.74
2.69
2.69
2.67
2.67
2.65
2.65
2.02
2.01
2.00
2.00
1.99
1.98
1.98
1.97
1.96
1.96
1.94
F
N
25
^{1}H NMR(400 MHz, CDCl$_3$)
0.85
1.79
2.75
0.92
0.88
4.00
2.04
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.
f1 (ppm)

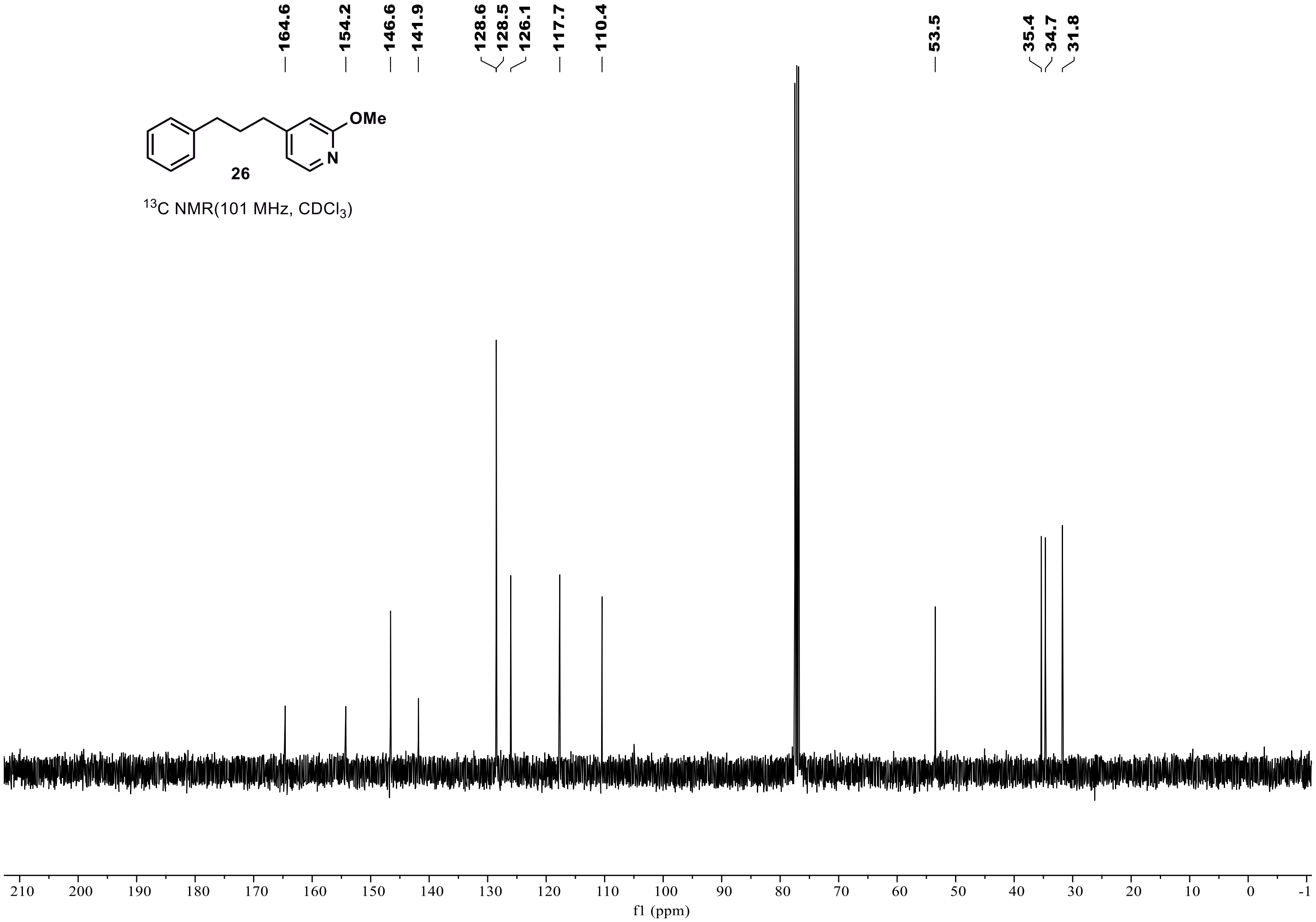


25

^{13}C NMR(101 MHz, $CDCl_3$)

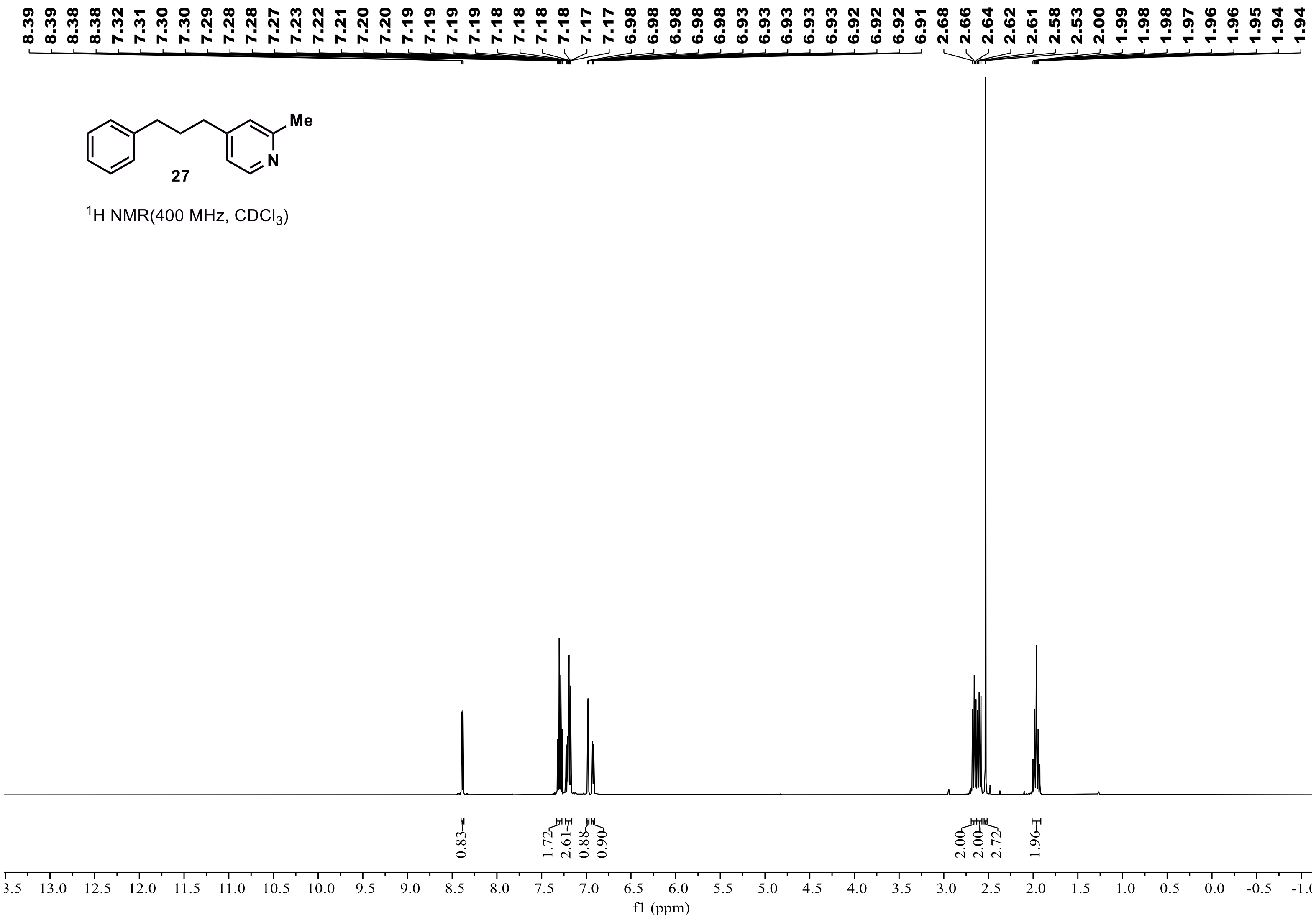

-69.17
F
N
25
^{19}F NMR(376 MHz, $CDCl_3$)
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

8.06
8.06
8.04
8.04
7.30
7.30
7.29
7.28
7.28
7.27
7.26
7.26
7.25
7.21
7.20
7.19
7.18
7.18
7.17
7.17
7.17
7.17
7.17
7.17
7.16
7.16
7.16
7.16
7.15
7.15
7.15
6.72
6.72
6.71
6.70
6.57
6.57
6.57
6.57
6.57
3.93
2.66
2.64
2.62
2.60
2.59
2.57
1.98
1.97
1.96
1.96
1.95
1.95
1.94
1.93
1.93
1.92
1.91
OMe
N
26
^{1}H NMR(400 MHz, CDCl$_3$)
0.85
1.73
2.65
0.89
0.83
2.79
2.00
2.00
2.00
3.5
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)

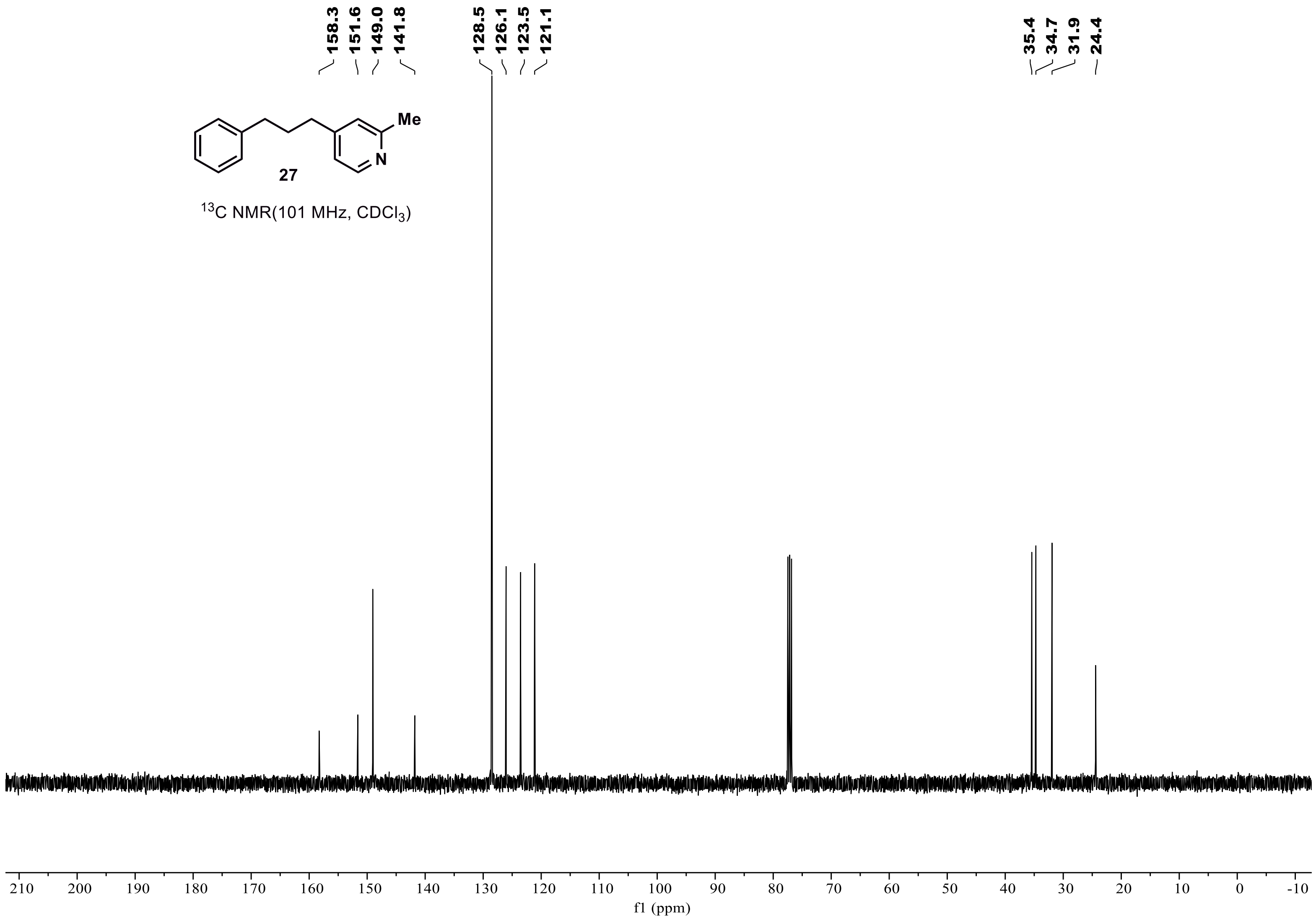


26

^{13}C NMR(101 MHz, $CDCl_3$)

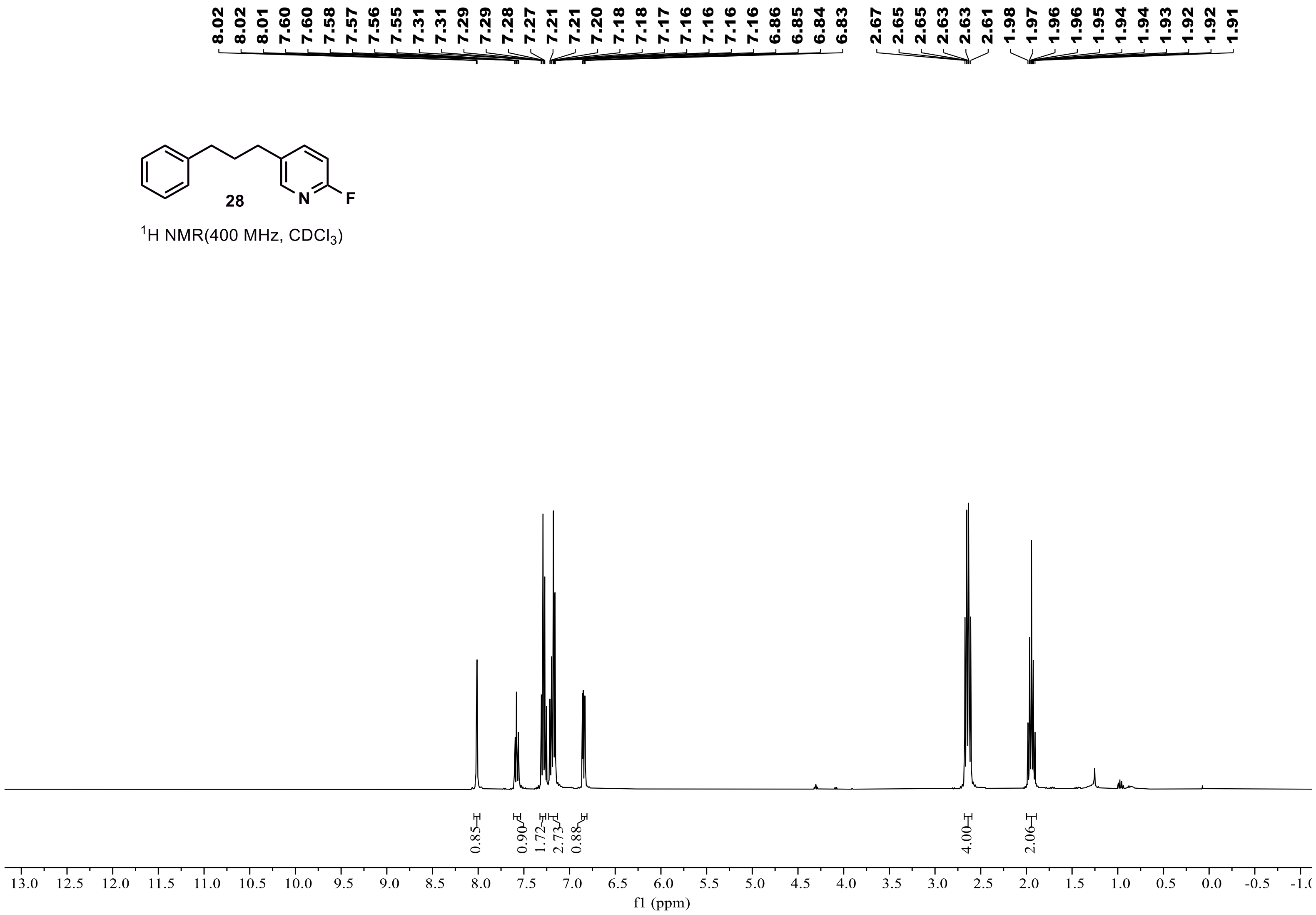


^{1}H NMR(400 MHz, $CDCl_3$)



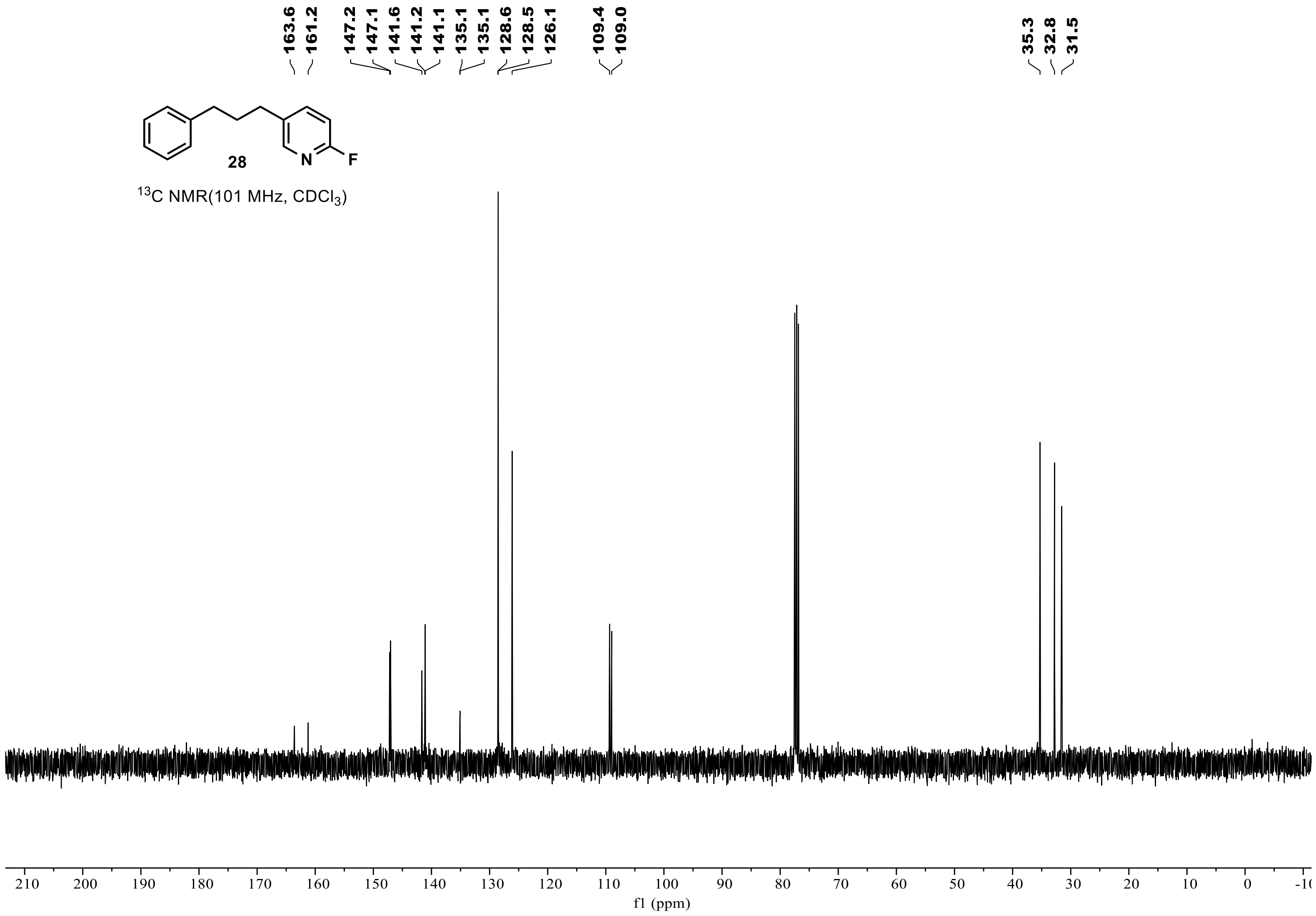
27

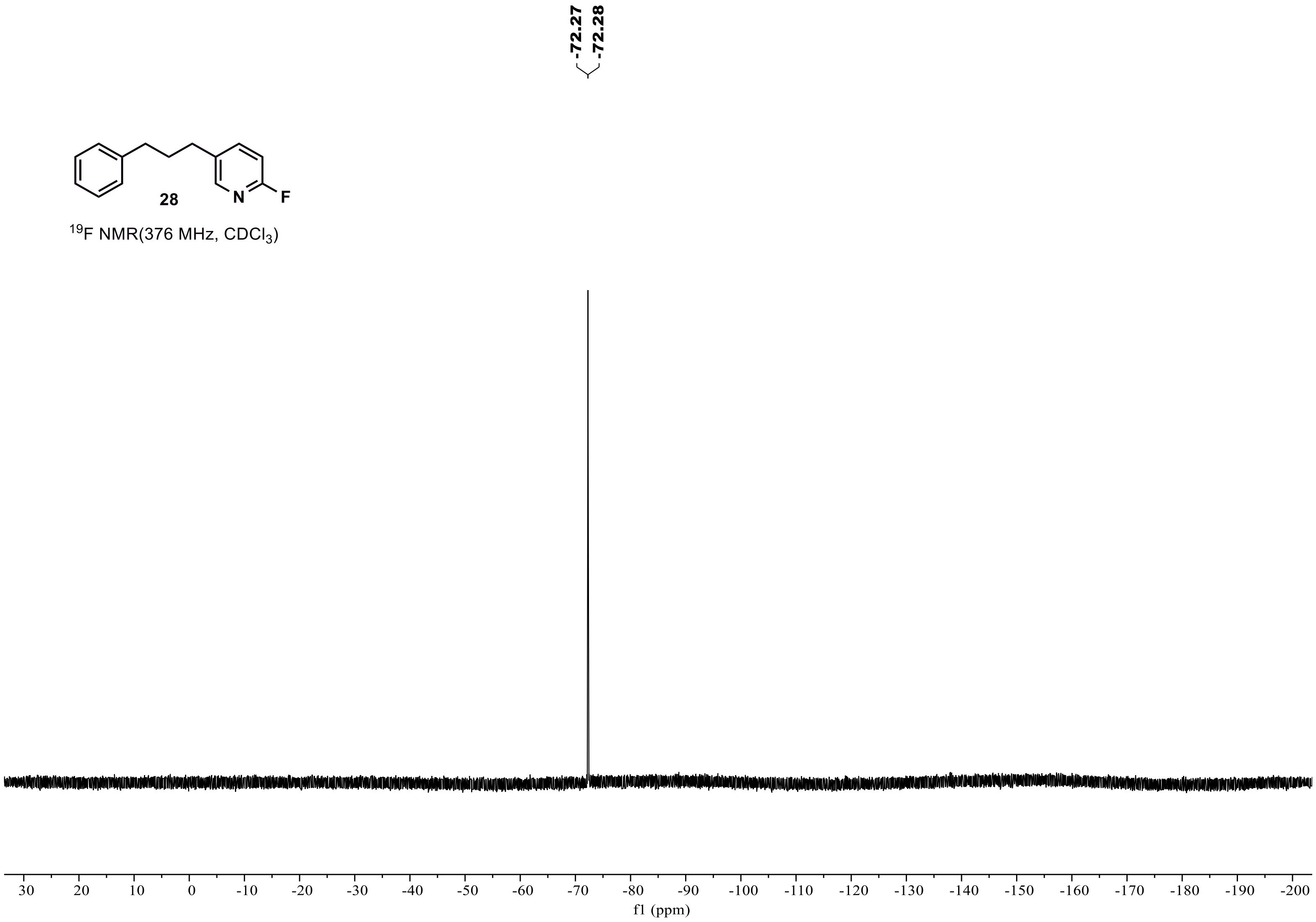
^{13}C NMR(101 MHz, CDCl$_3$)



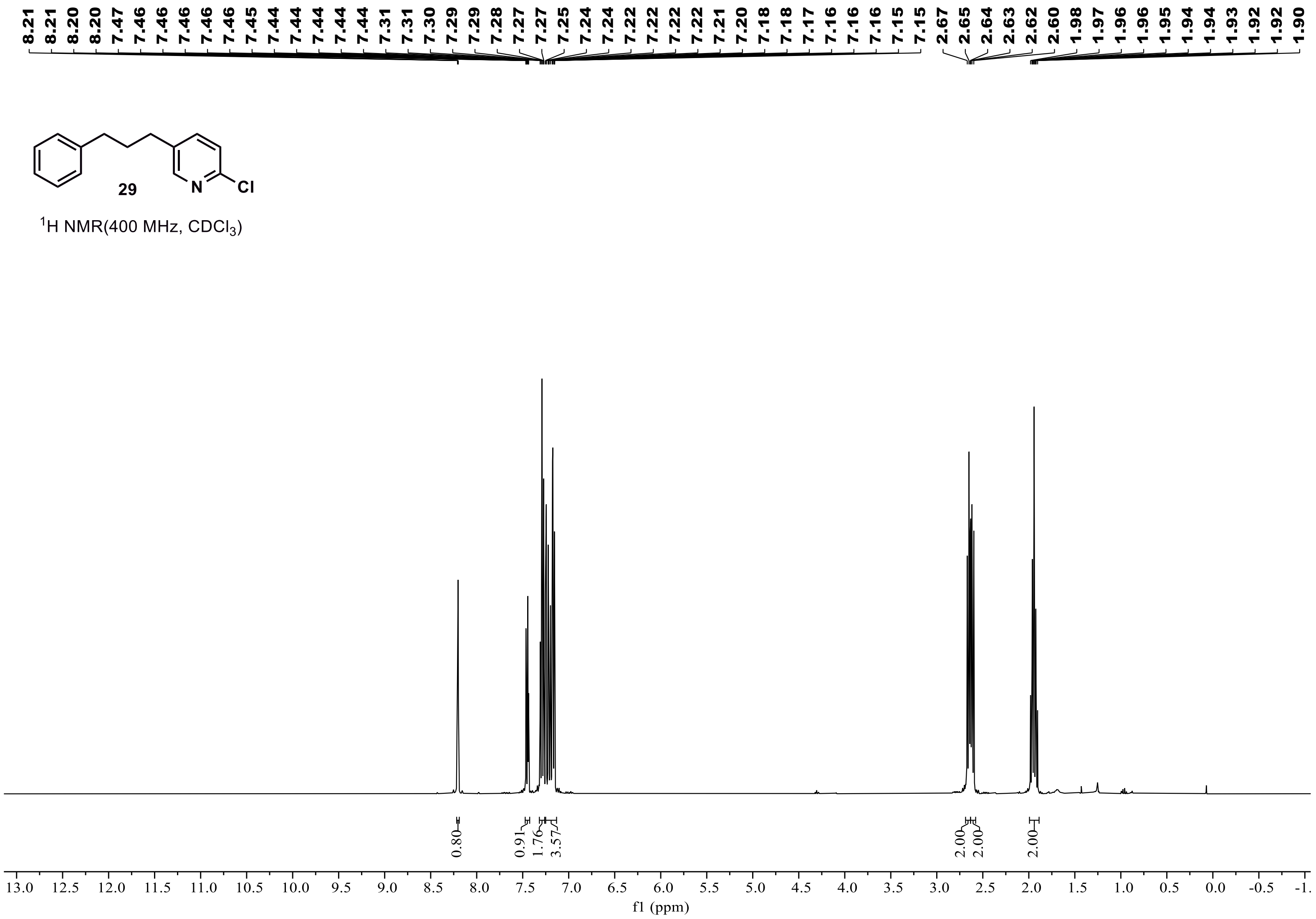
28

^{1}H NMR(400 MHz, $CDCl_3$)


163.6
161.2
147.2
147.1
141.6
141.2
141.1
135.1
135.1
128.6
128.5
126.1
109.4
109.0
35.3
32.8
31.5
N
F
28
^{13}C NMR(101 MHz, CDCl$_3$)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

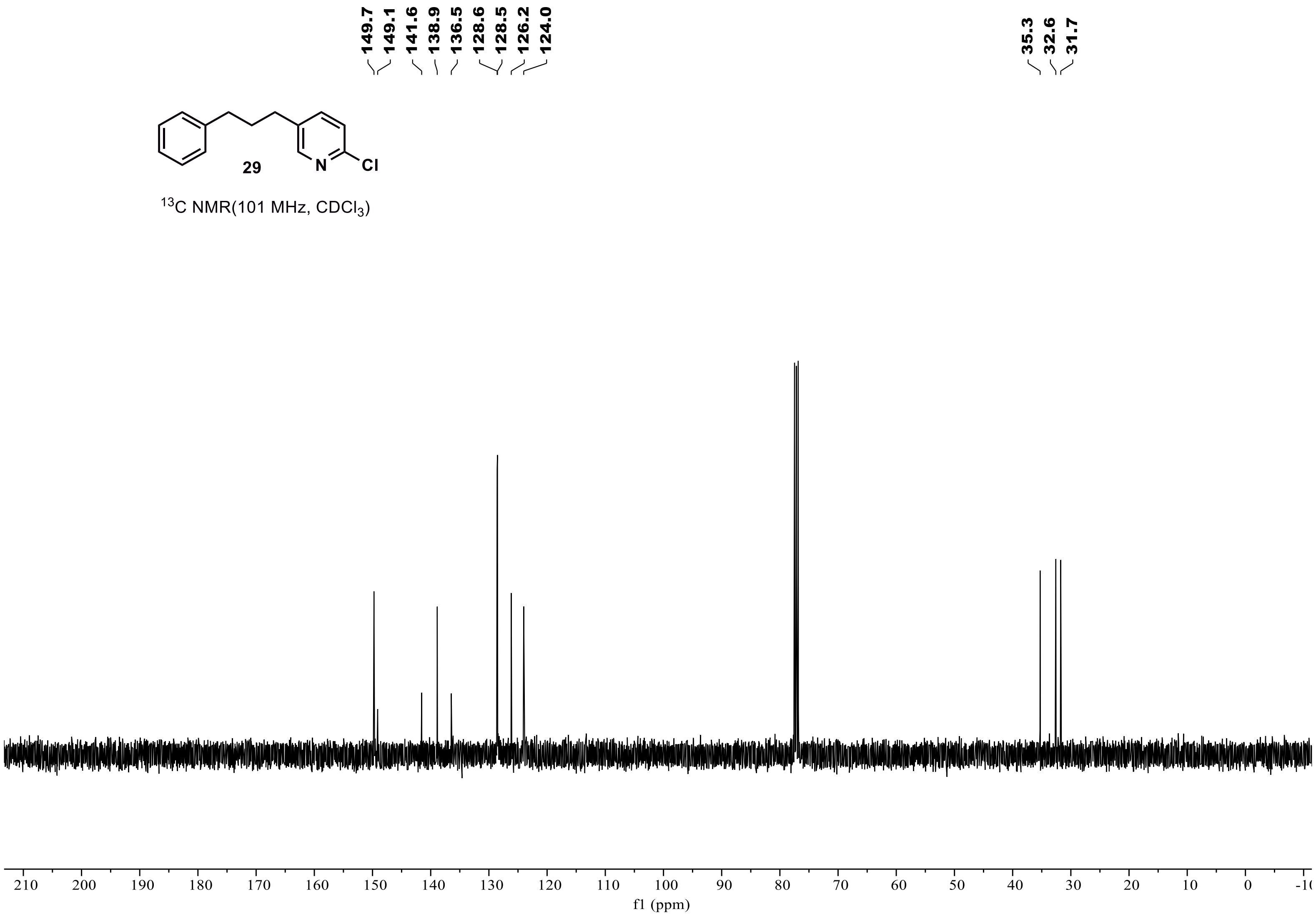

28

^{19}F NMR(376 MHz, $CDCl_3$)



29

^{1}H NMR(400 MHz, $CDCl_3$)

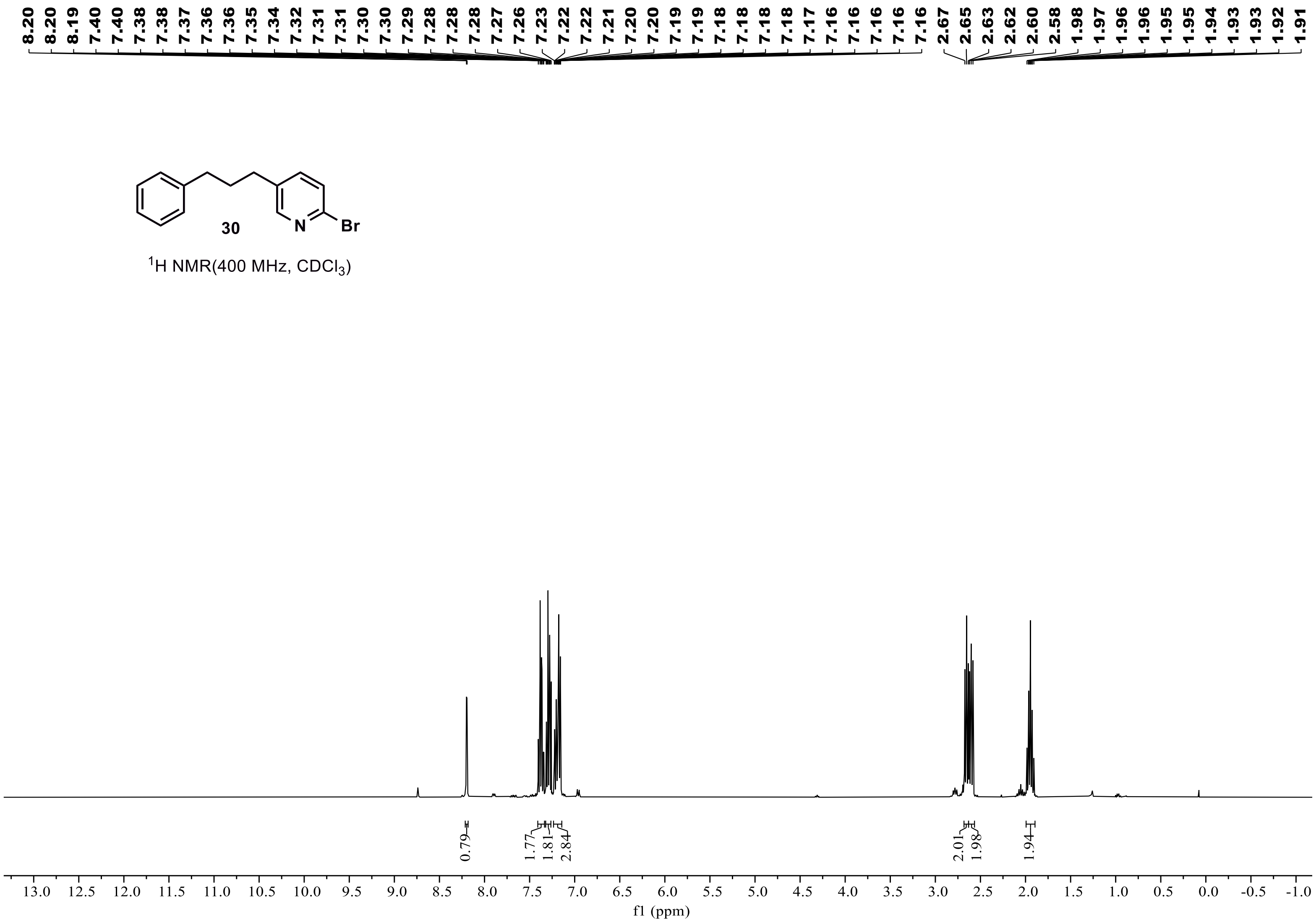


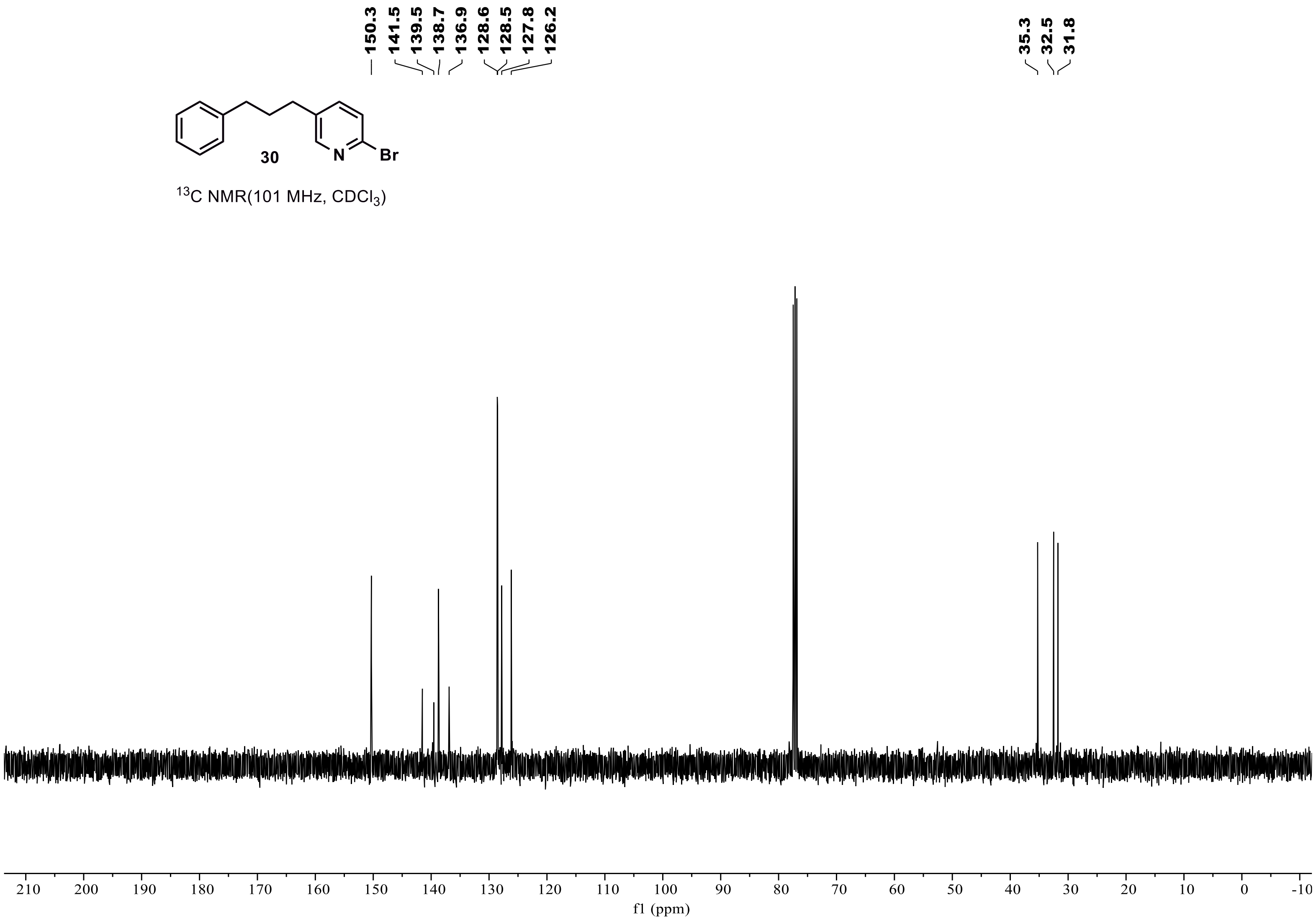
29

^{13}C NMR(101 MHz, $CDCl_3$)

30

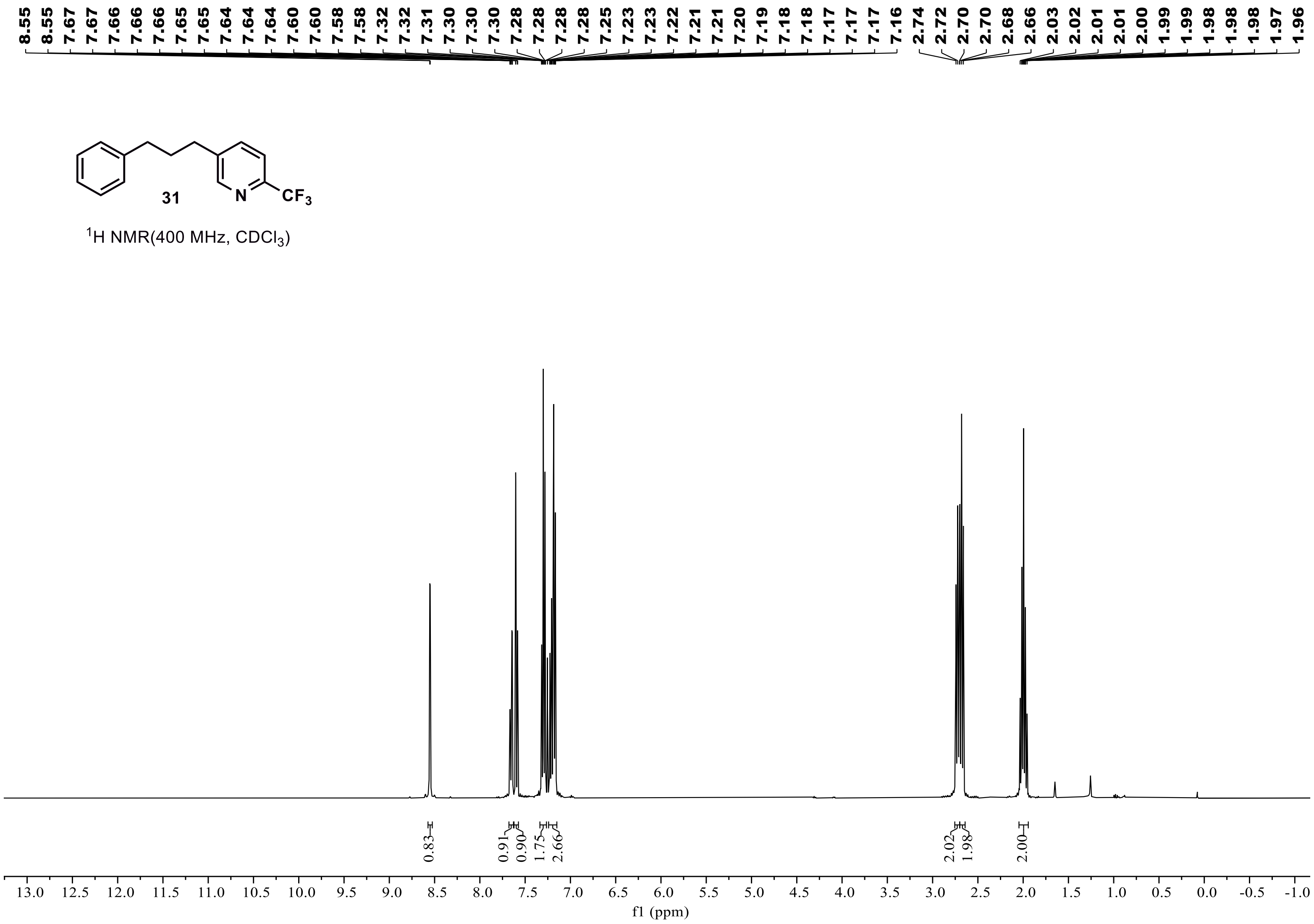
^{1}H NMR(400 MHz, CDCl$_3$)





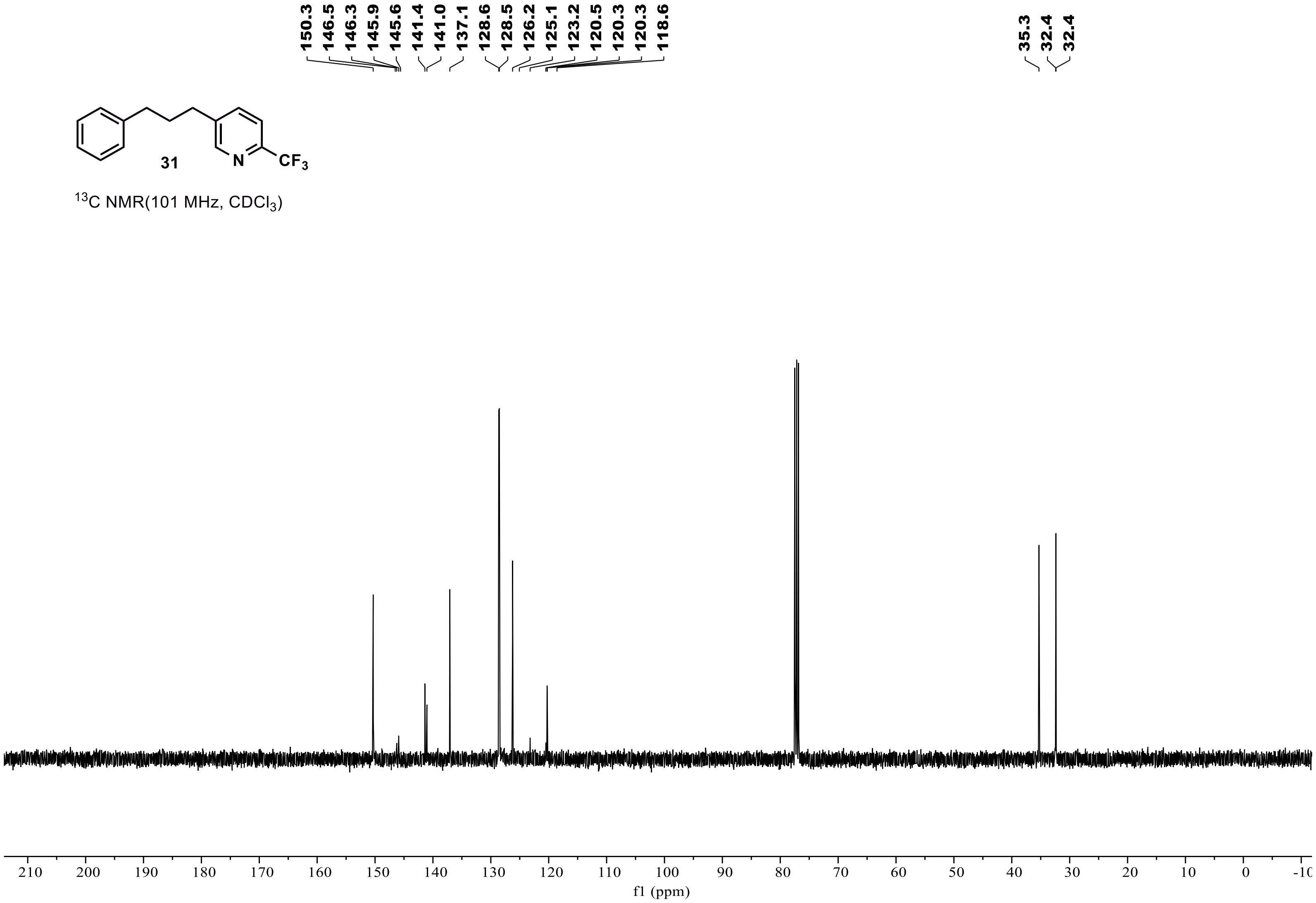
30

^{13}C NMR(101 MHz, CDCl$_3$)



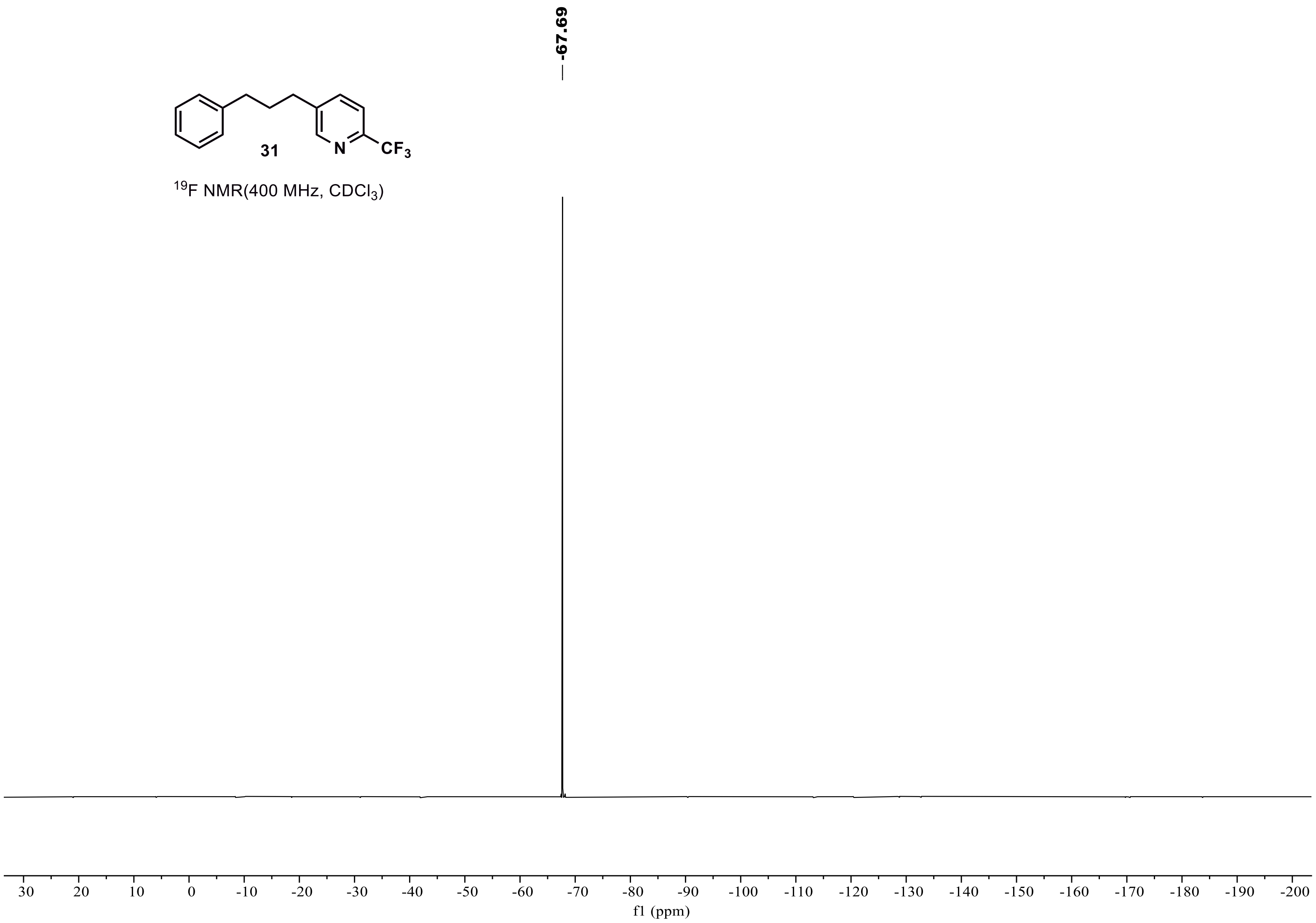
31

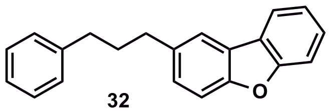
^{1}H NMR(400 MHz, $CDCl_3$)



31

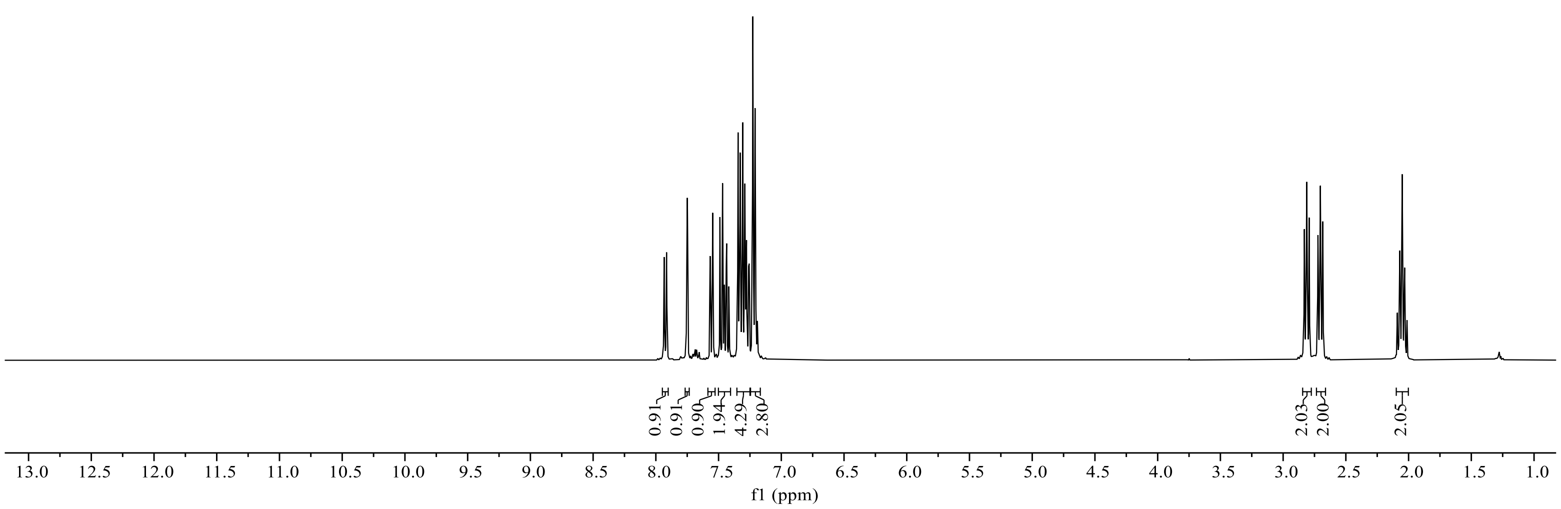
^{13}C NMR(101 MHz, $CDCl_3$)

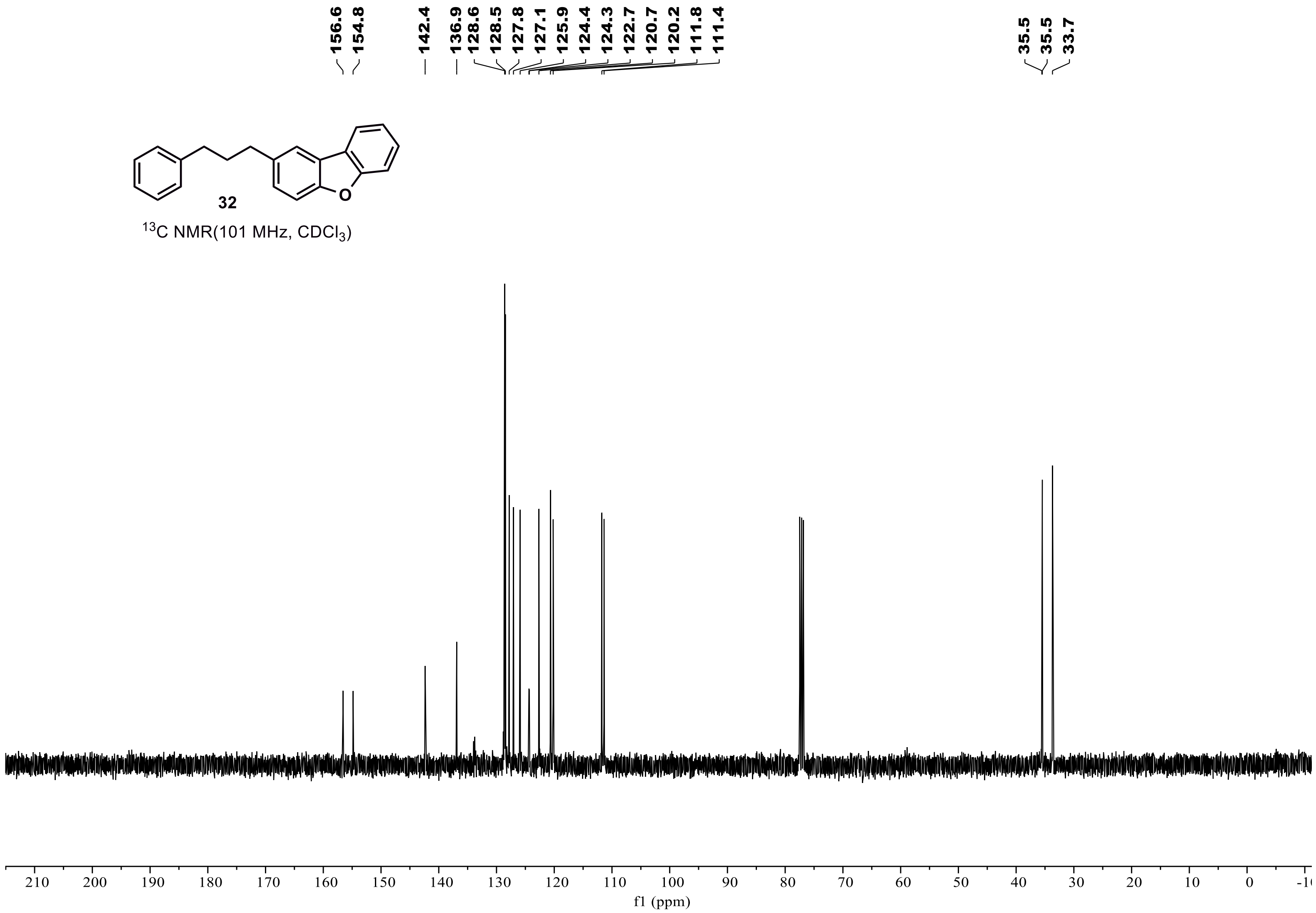

-67.69
31
N
CF3
19F NMR(400 MHz, CDCl3)
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)



32

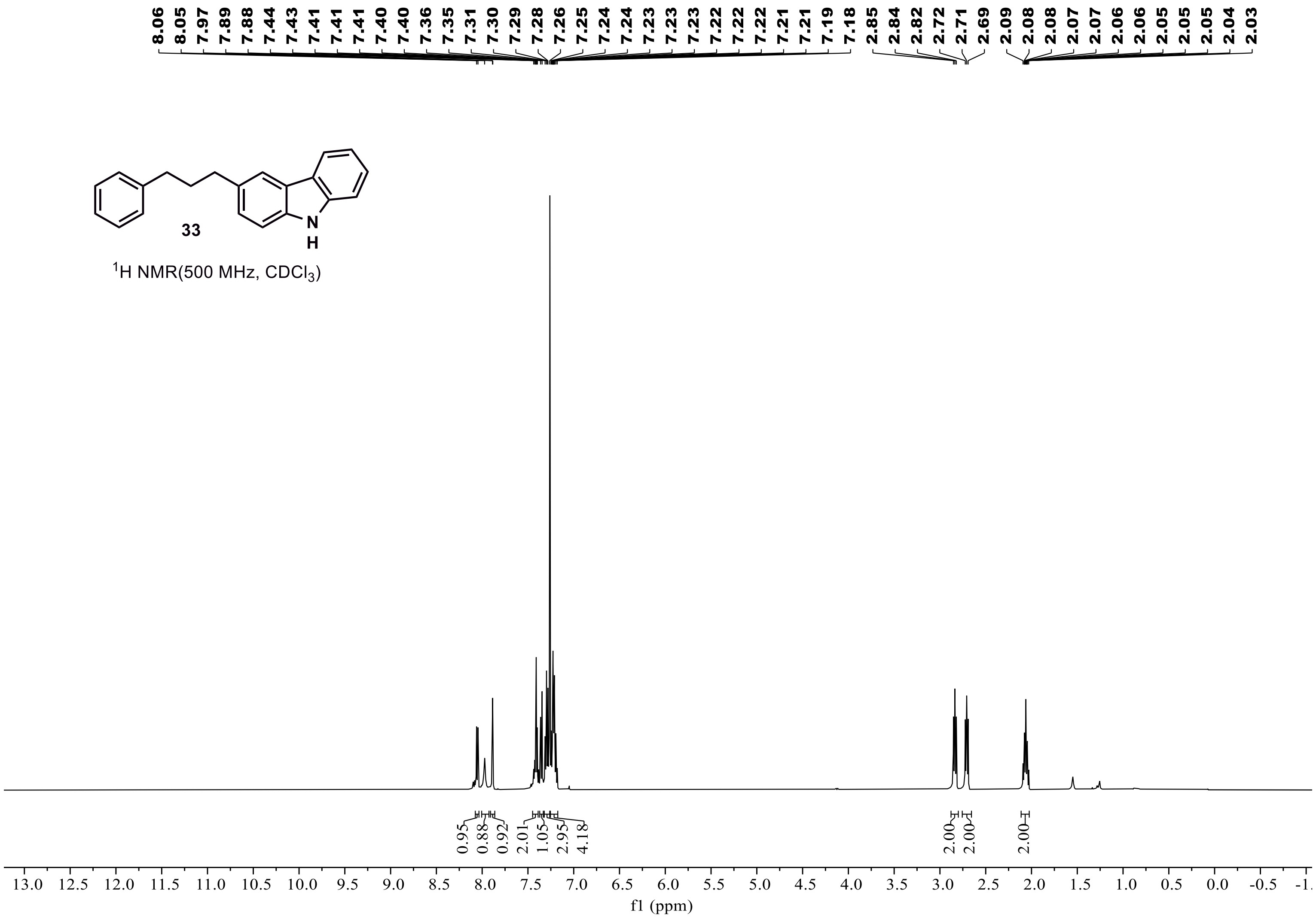
^{1}H NMR(400 MHz, CDCl$_3$)





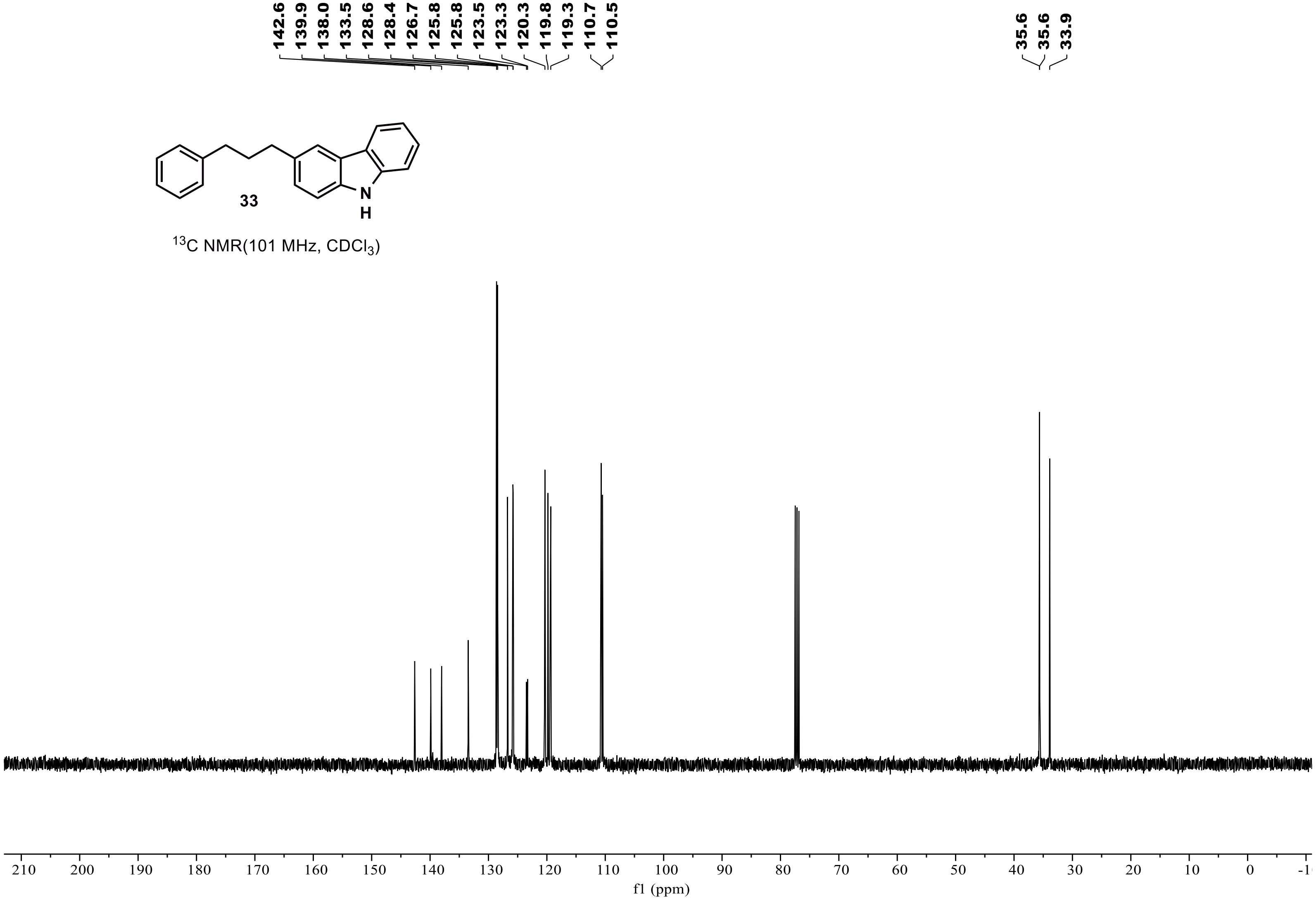
32

^{13}C NMR(101 MHz, CDCl$_3$)



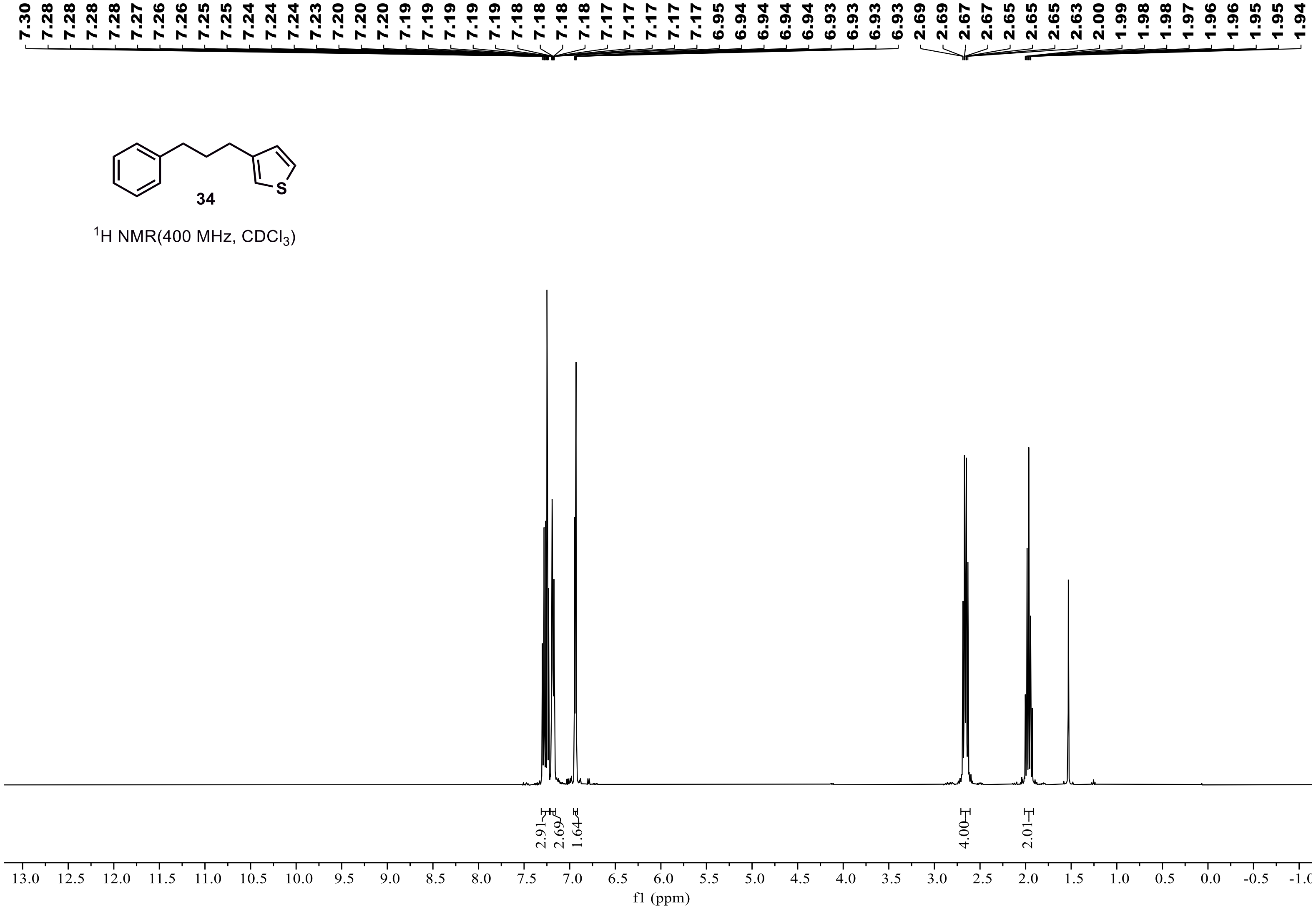
33

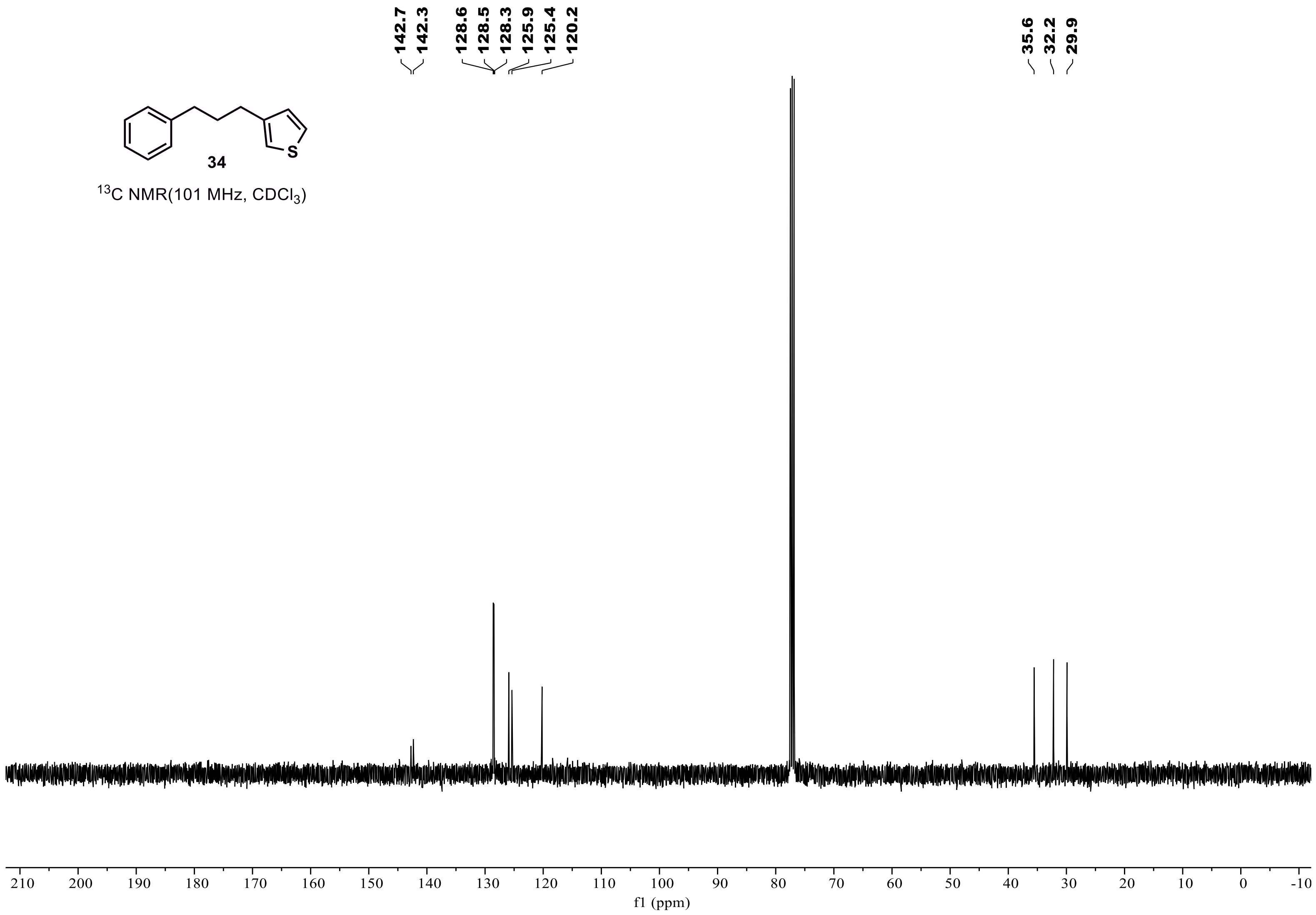
^{1}H NMR(500 MHz, $CDCl_3$)



33

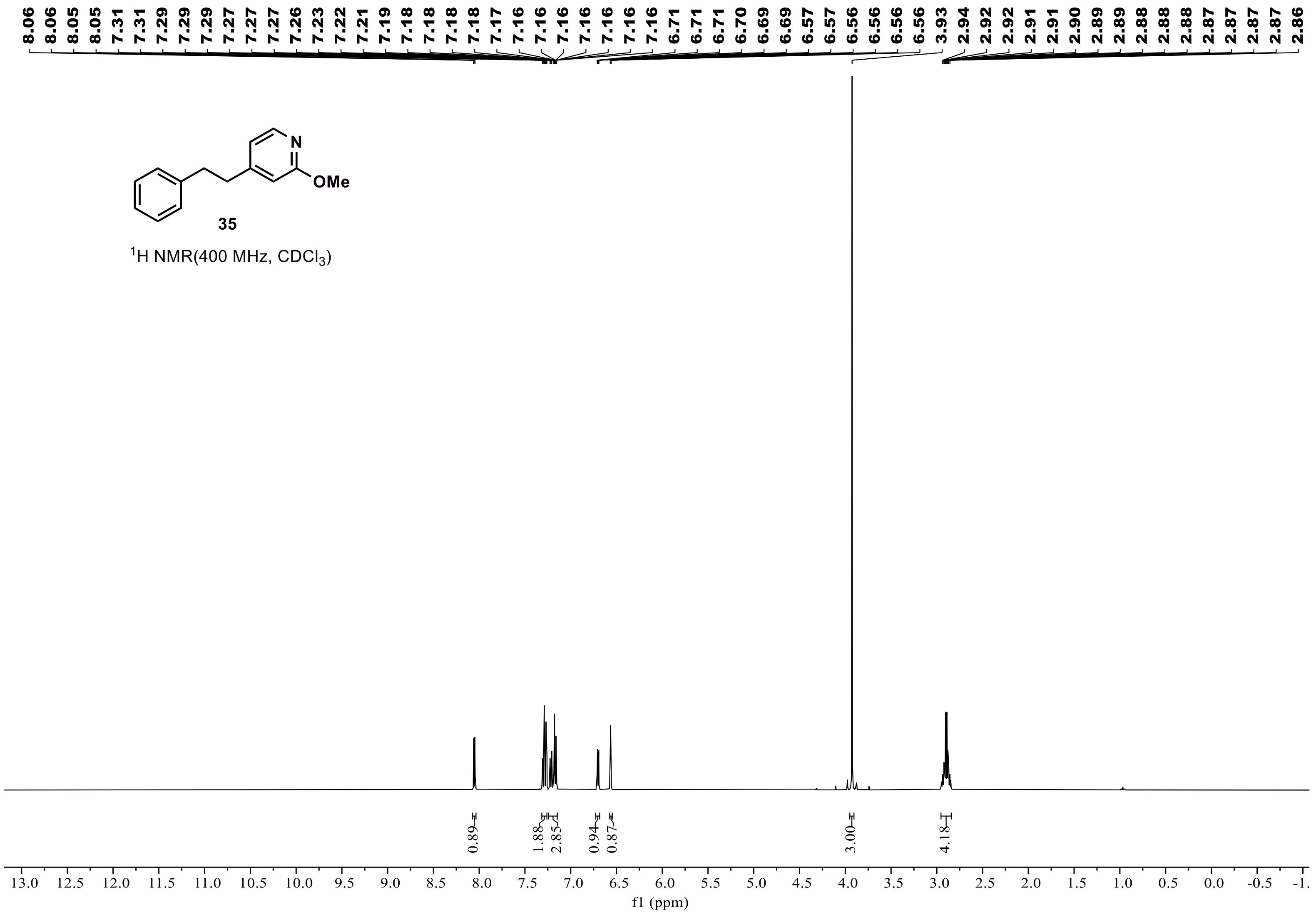
^{13}C NMR(101 MHz, CDCl$_3$)

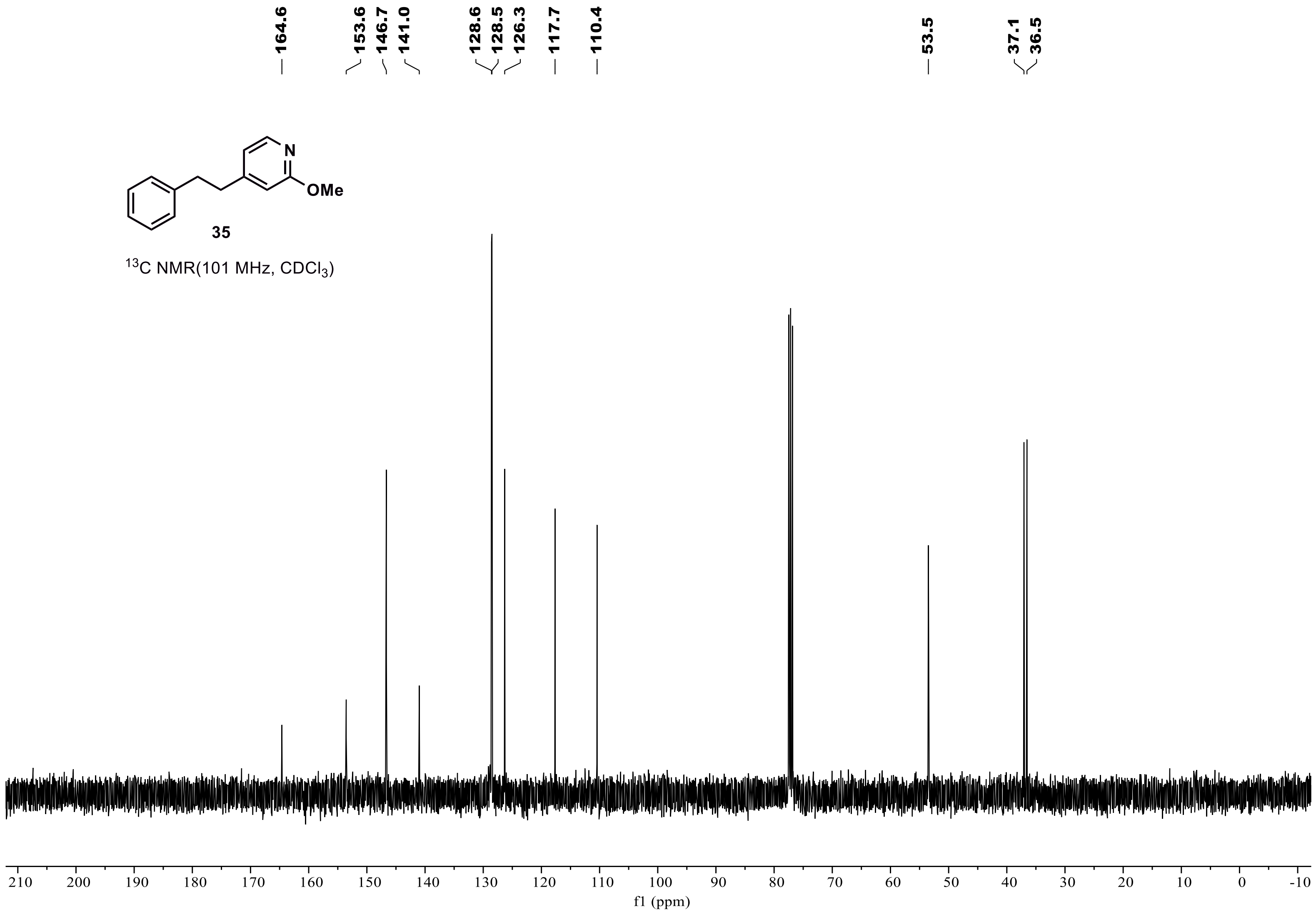

7.30 7.28 7.28 7.28 7.28 7.27 7.26 7.26 7.25 7.25 7.24 7.24 7.24 7.23 7.20 7.20 7.19 7.19 7.19 7.19 7.19 7.18 7.18 7.18 7.18 7.17 7.17 7.17 7.17 7.17 6.95 6.94 6.94 6.94 6.94 6.93 6.93 6.93 6.93 2.69 2.69 2.67 2.67 2.65 2.65 2.65 2.63 2.00 1.99 1.98 1.98 1.97 1.96 1.96 1.95 1.95 1.94
S
34
^{1}H NMR(400 MHz, CDCl$_3$)
2.91
2.69
1.64
4.00
2.01
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)



34

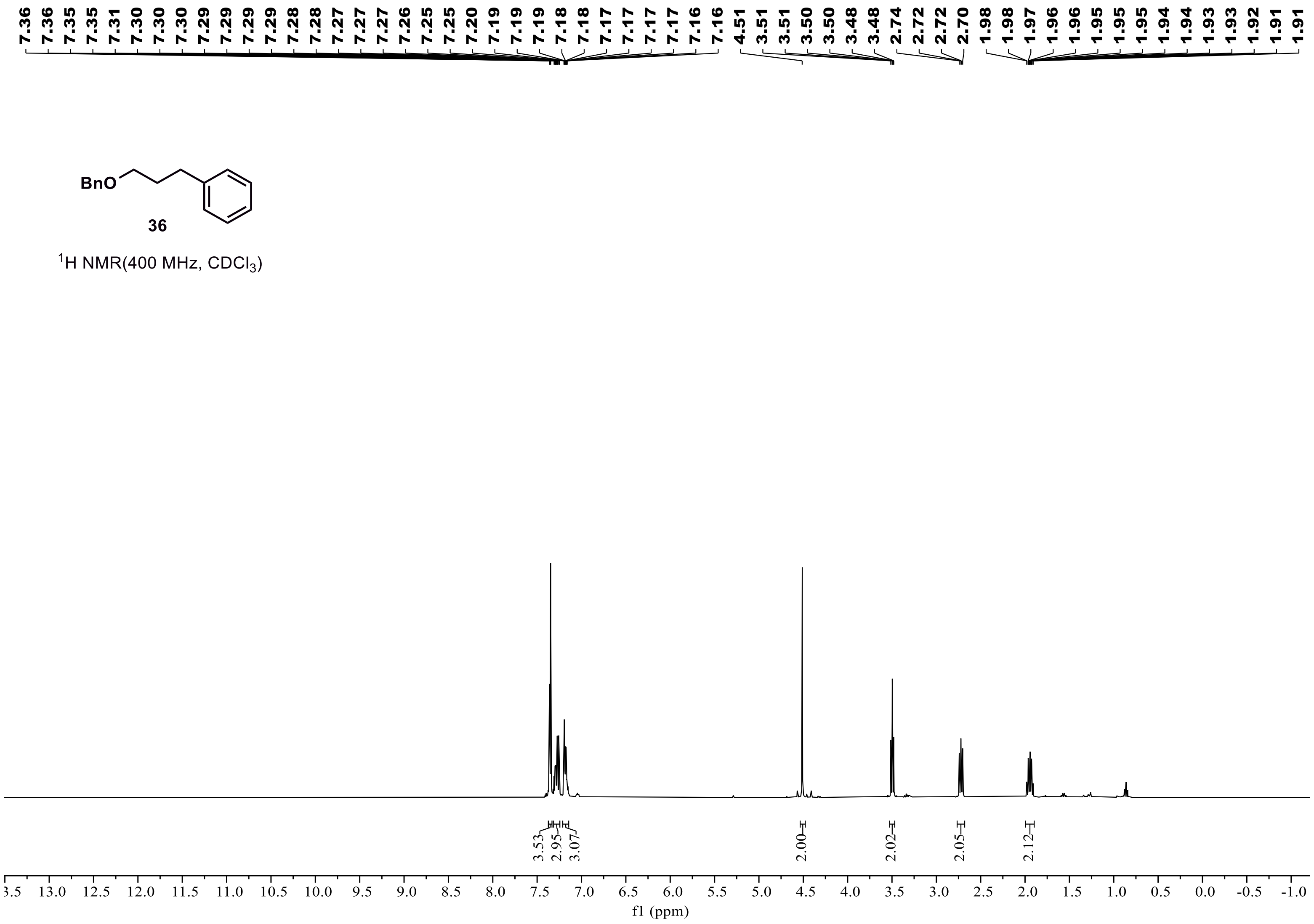
^{13}C NMR(101 MHz, CDCl$_3$)


8.06
8.06
8.05
8.05
7.31
7.31
7.29
7.29
7.29
7.27
7.27
7.27
7.26
7.23
7.22
7.21
7.19
7.18
7.18
7.18
7.18
7.17
7.16
7.16
7.16
7.16
7.16
7.16
6.71
6.71
6.71
6.70
6.69
6.69
6.57
6.57
6.56
6.56
6.56
6.56
3.93
2.94
2.92
2.92
2.91
2.91
2.90
2.89
2.89
2.88
2.88
2.88
2.87
2.87
2.87
2.87
2.86
N
OMe
35
^{1}H NMR(400 MHz, CDCl$_3$)
0.89
1.88
2.85
0.94
0.87
3.00
4.18
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.
f1 (ppm)



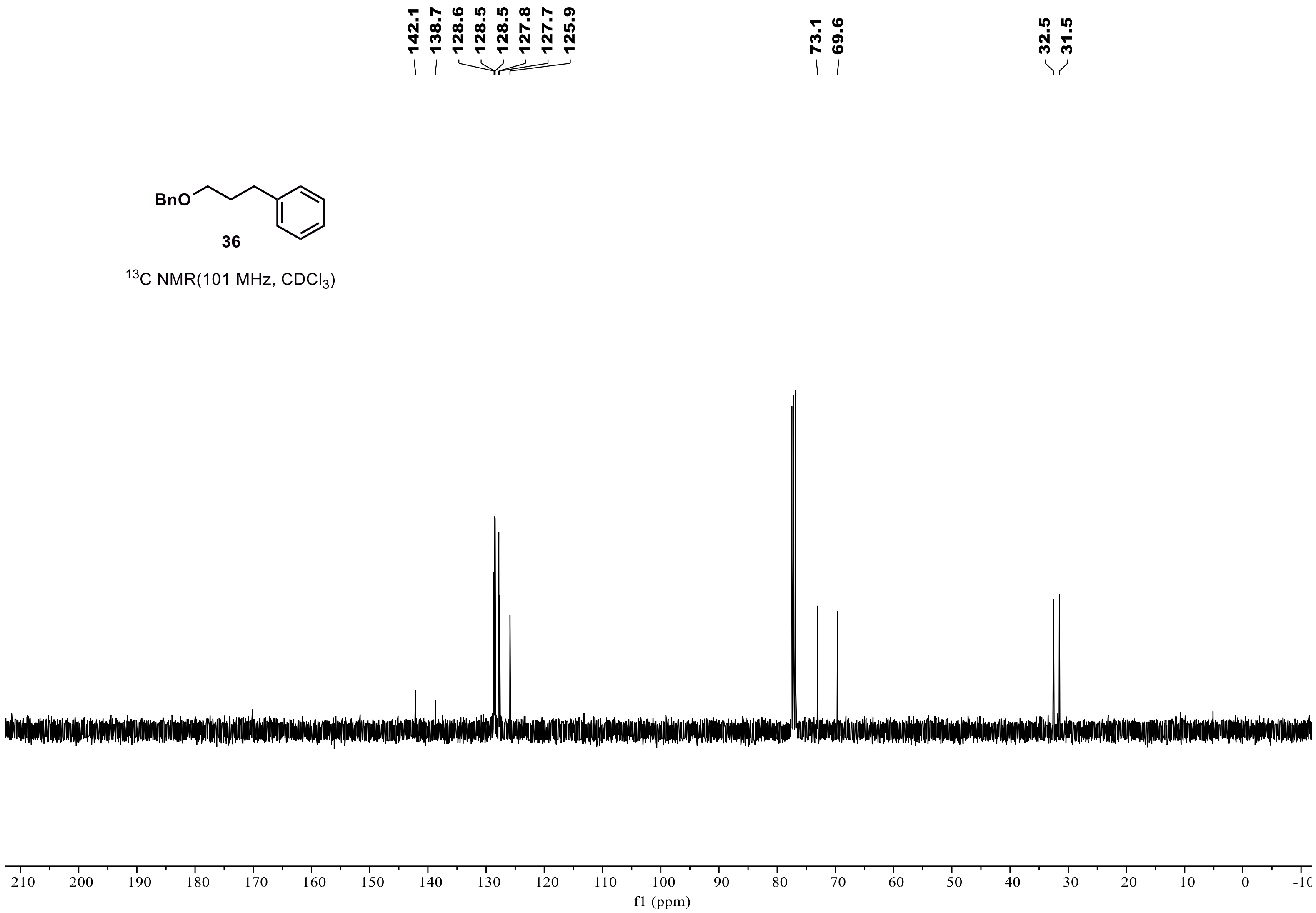
35

^{13}C NMR(101 MHz, CDCl$_3$)



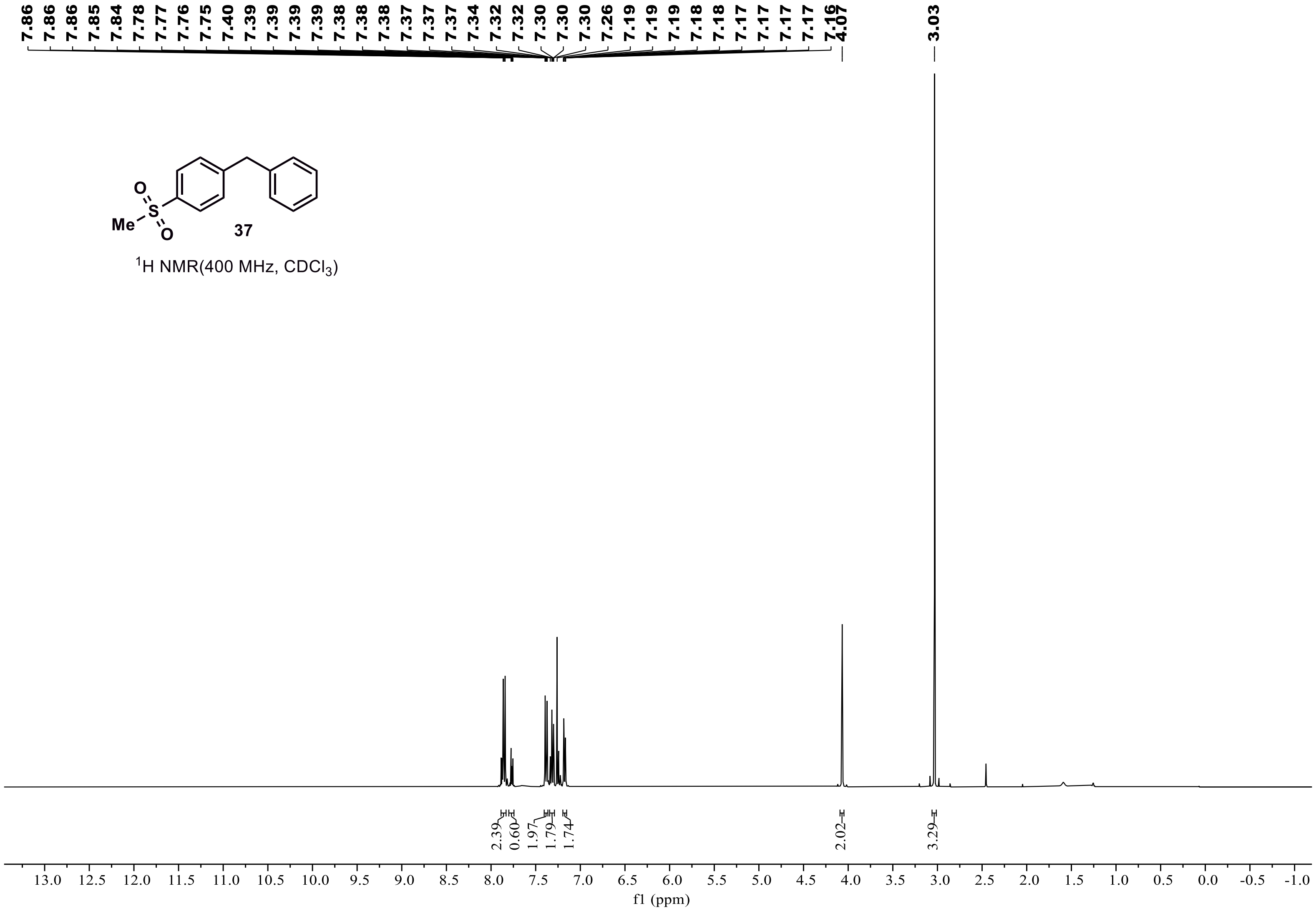
36

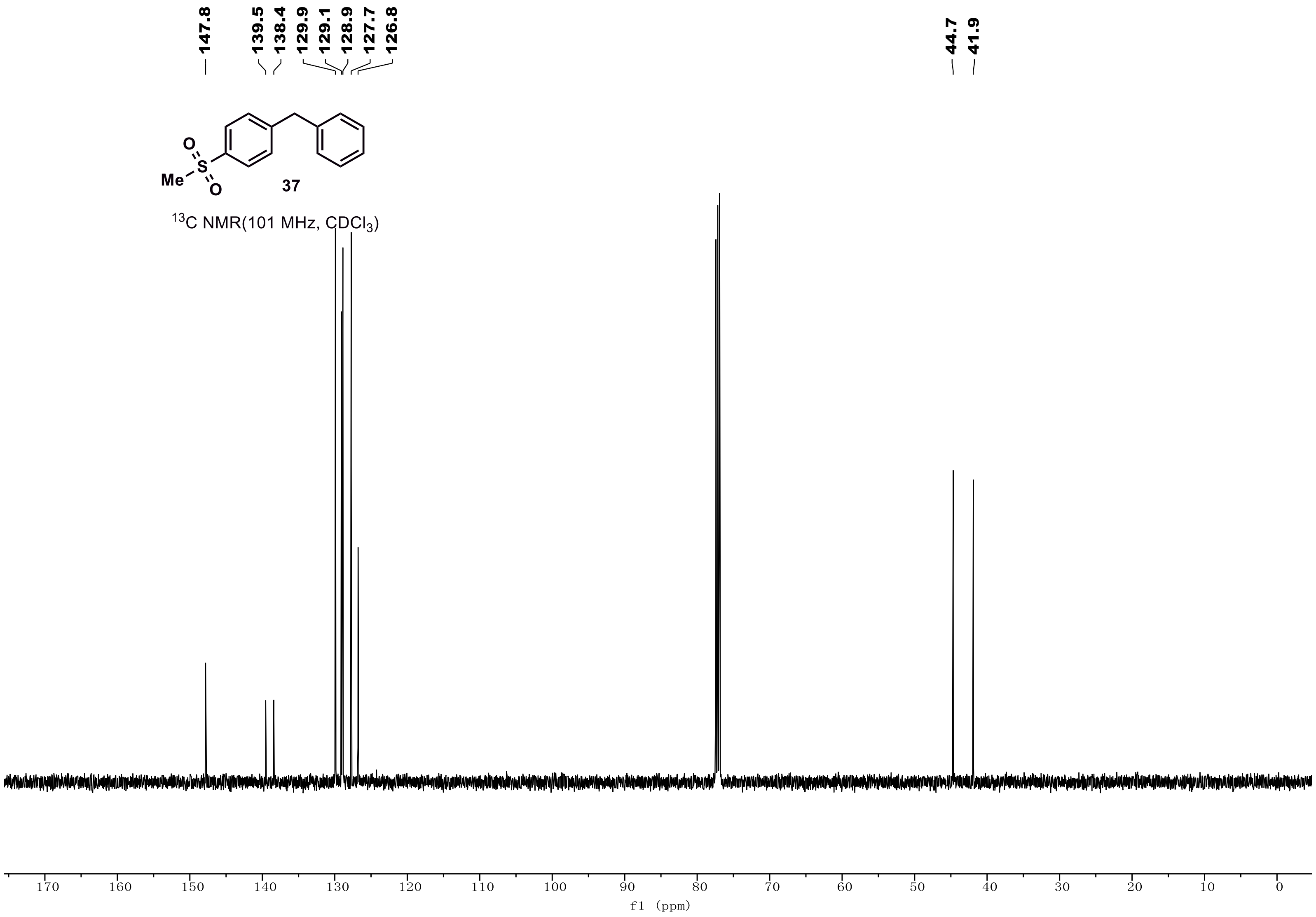
^{1}H NMR(400 MHz, $CDCl_3$)

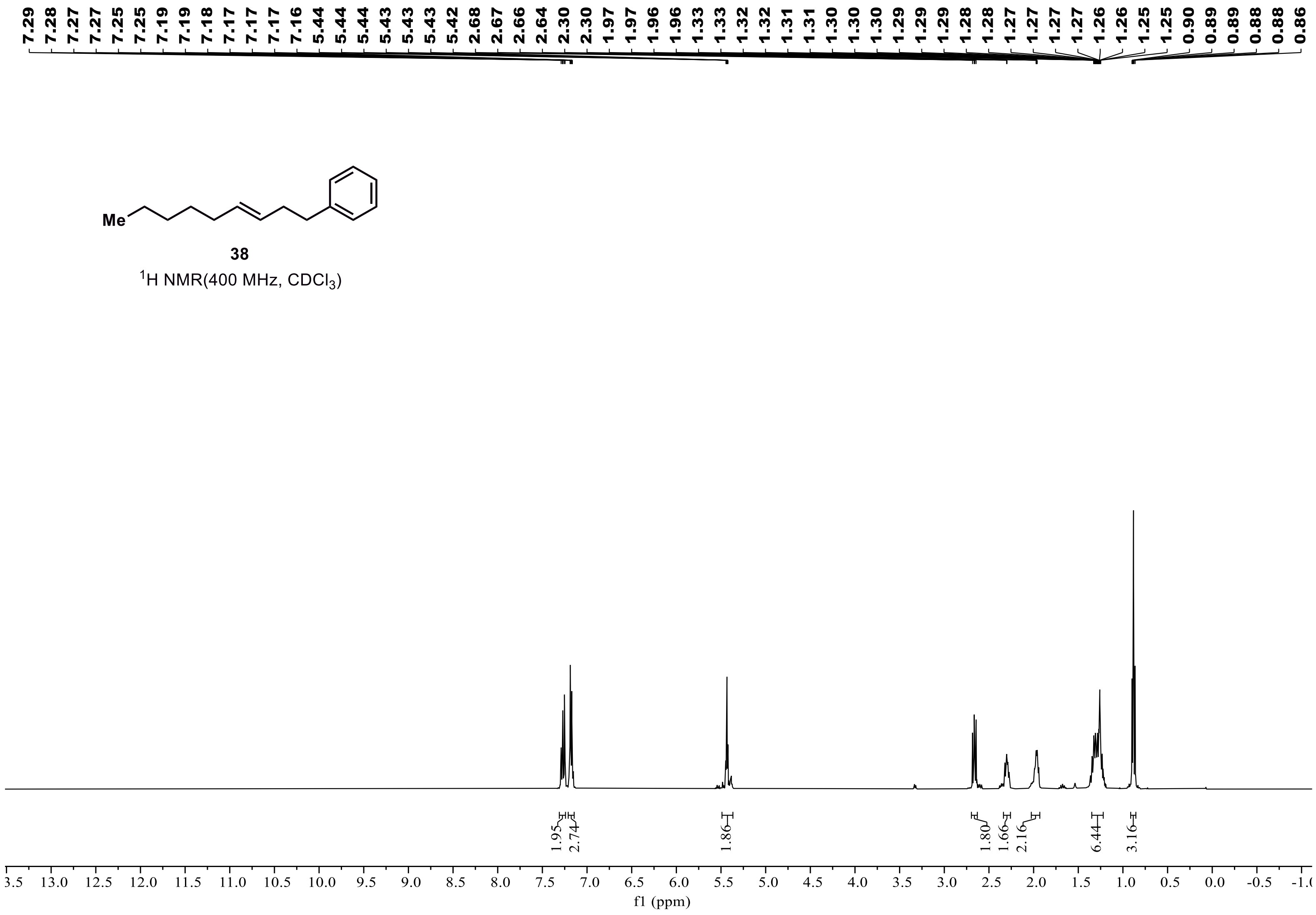


36

^{13}C NMR(101 MHz, CDCl$_3$)

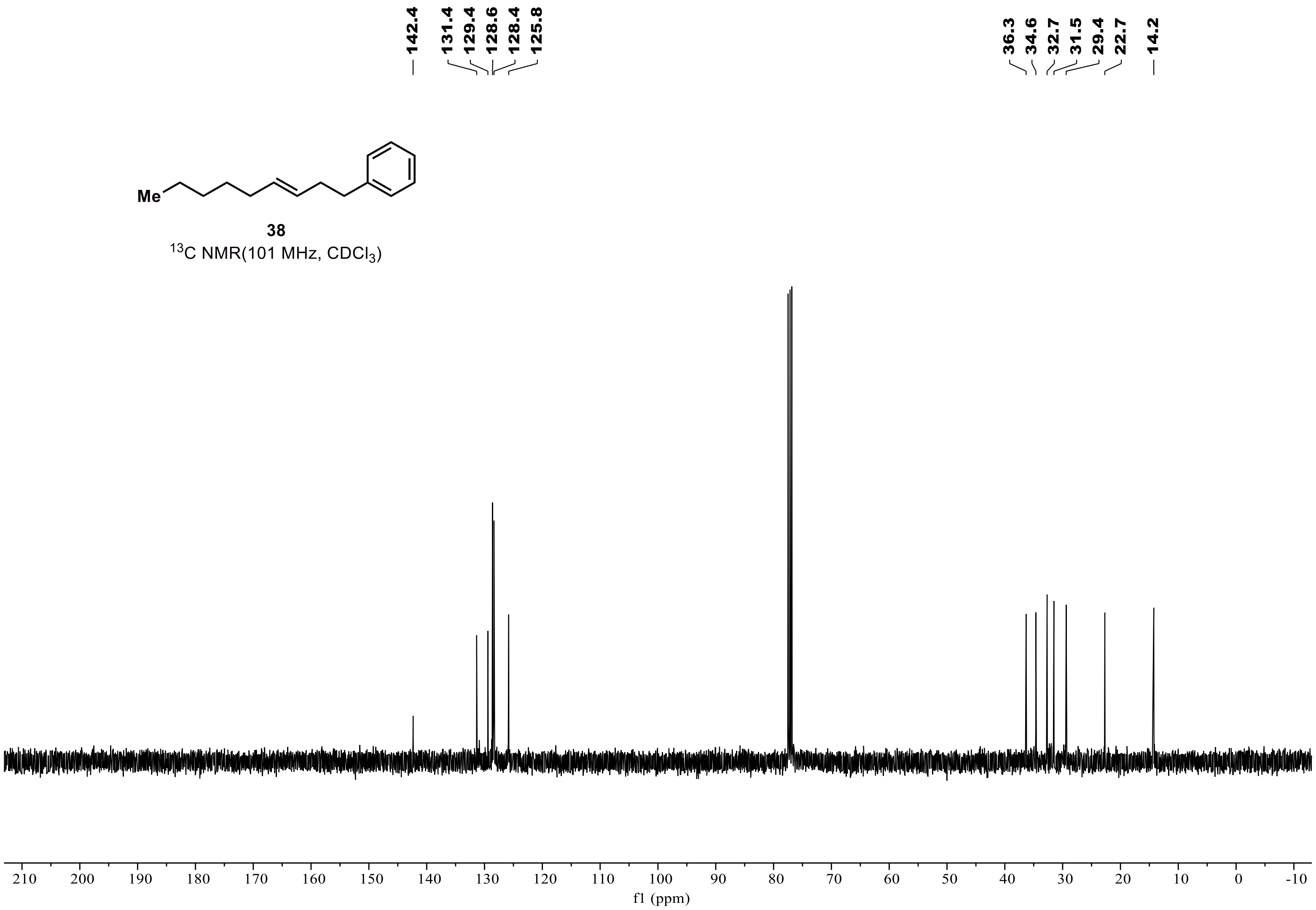

7.86
7.86
7.86
7.85
7.84
7.78
7.77
7.76
7.75
7.40
7.39
7.39
7.39
7.39
7.38
7.38
7.38
7.37
7.37
7.34
7.32
7.32
7.30
7.30
7.30
7.26
7.19
7.19
7.19
7.18
7.18
7.17
7.17
7.17
7.17
7.16
4.07
3.03
O
S
O
Me
37
^{1}H NMR(400 MHz, CDCl$_3$)
2.39
0.60
1.97
1.79
1.74
2.02
3.29
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


147.8
139.5
138.4
129.9
129.1
128.9
127.7
126.8
44.7
41.9
O
S
O
Me
37
13C NMR(101 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



38

^{1}H NMR(400 MHz, CDCl$_3$)

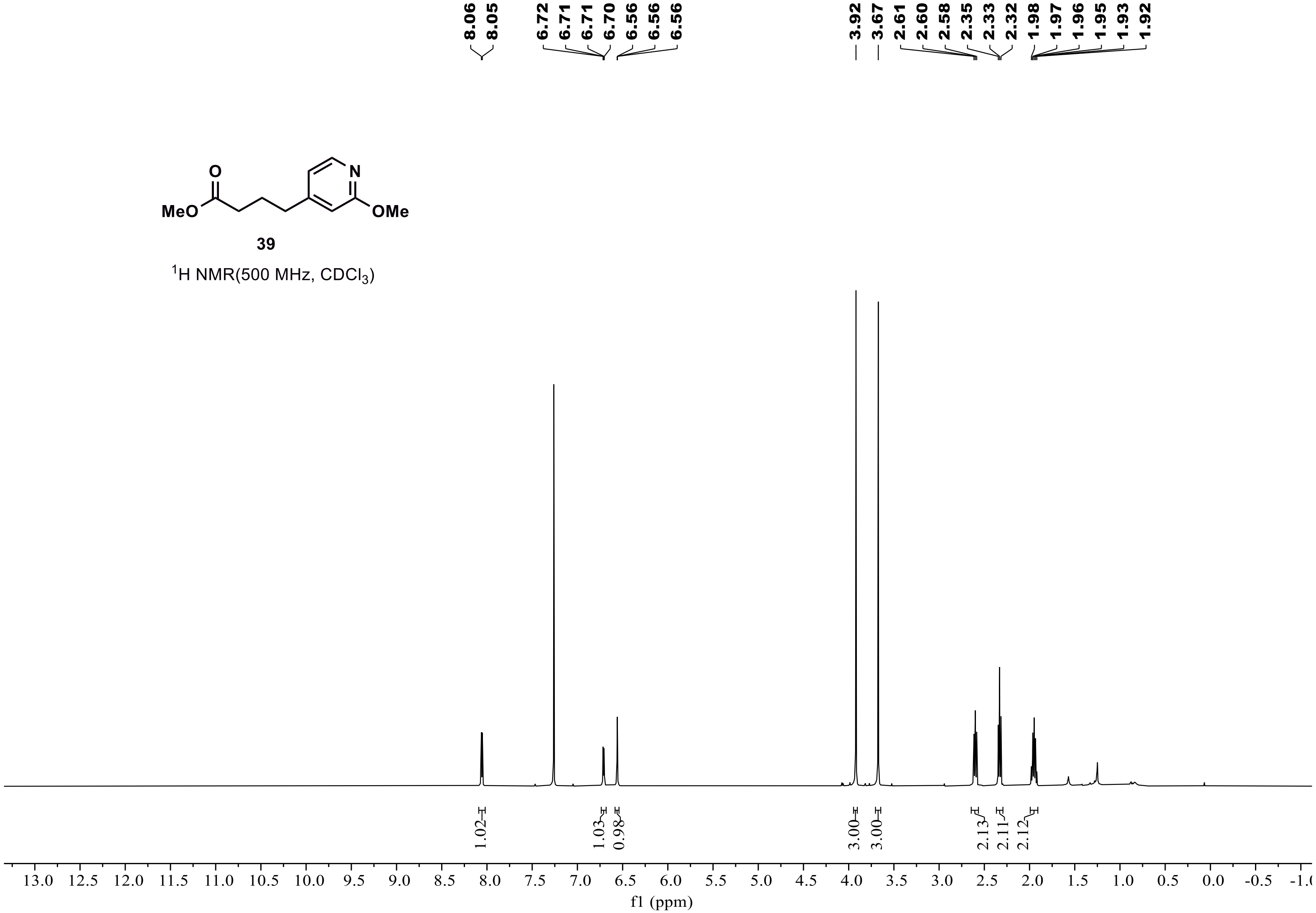


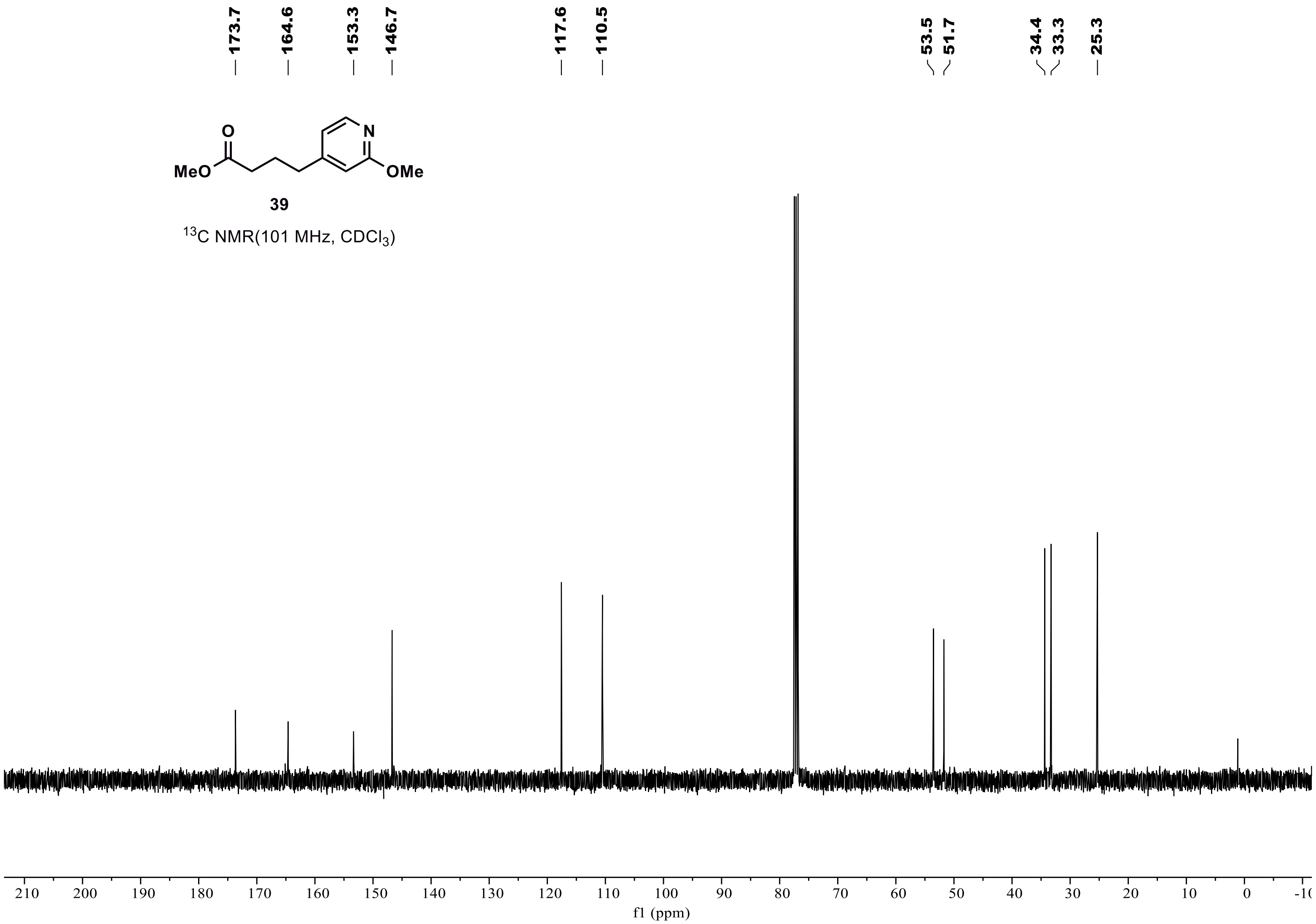
38

^{13}C NMR(101 MHz, CDCl$_3$)

39

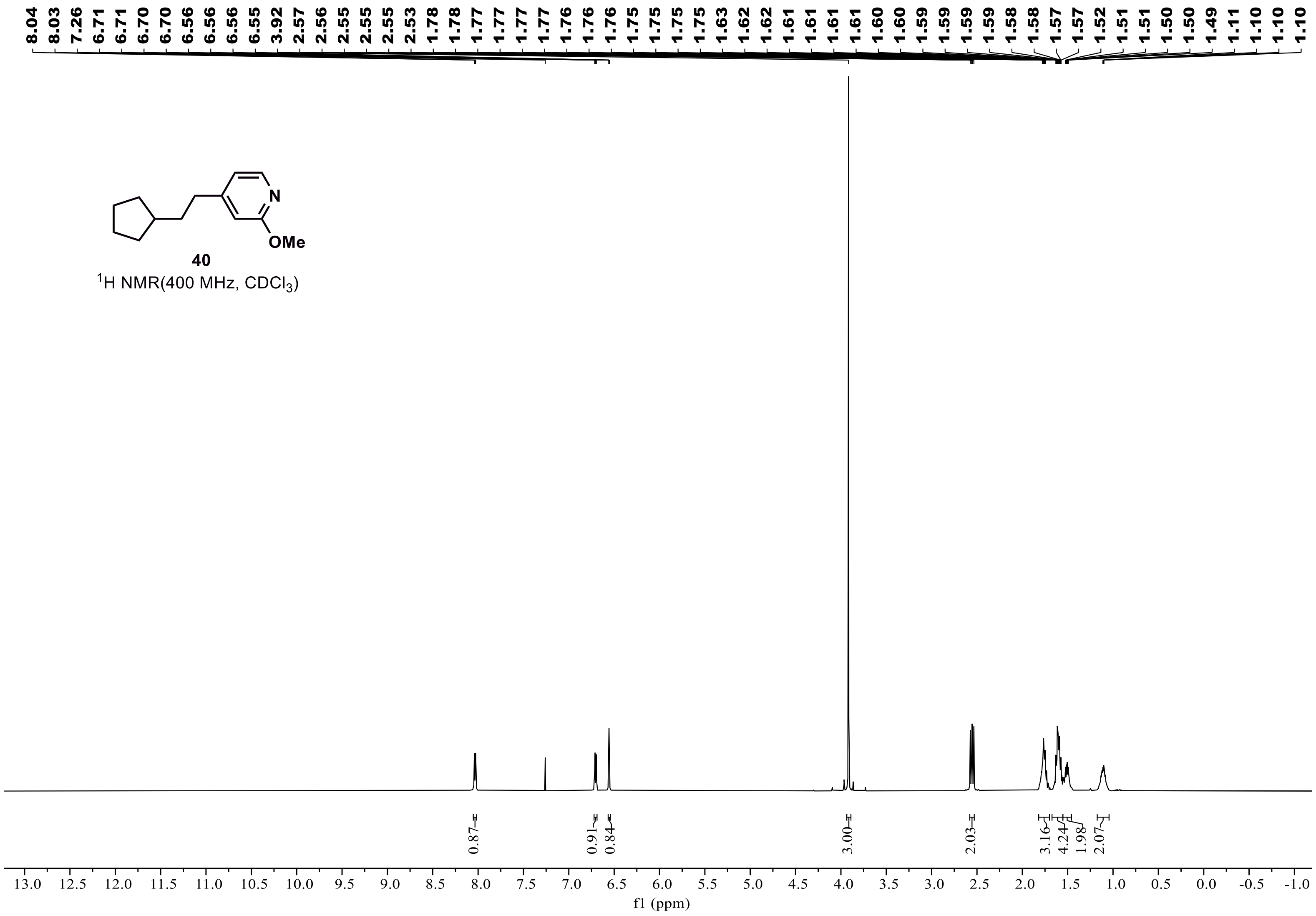
^{1}H NMR(500 MHz, CDCl$_3$)

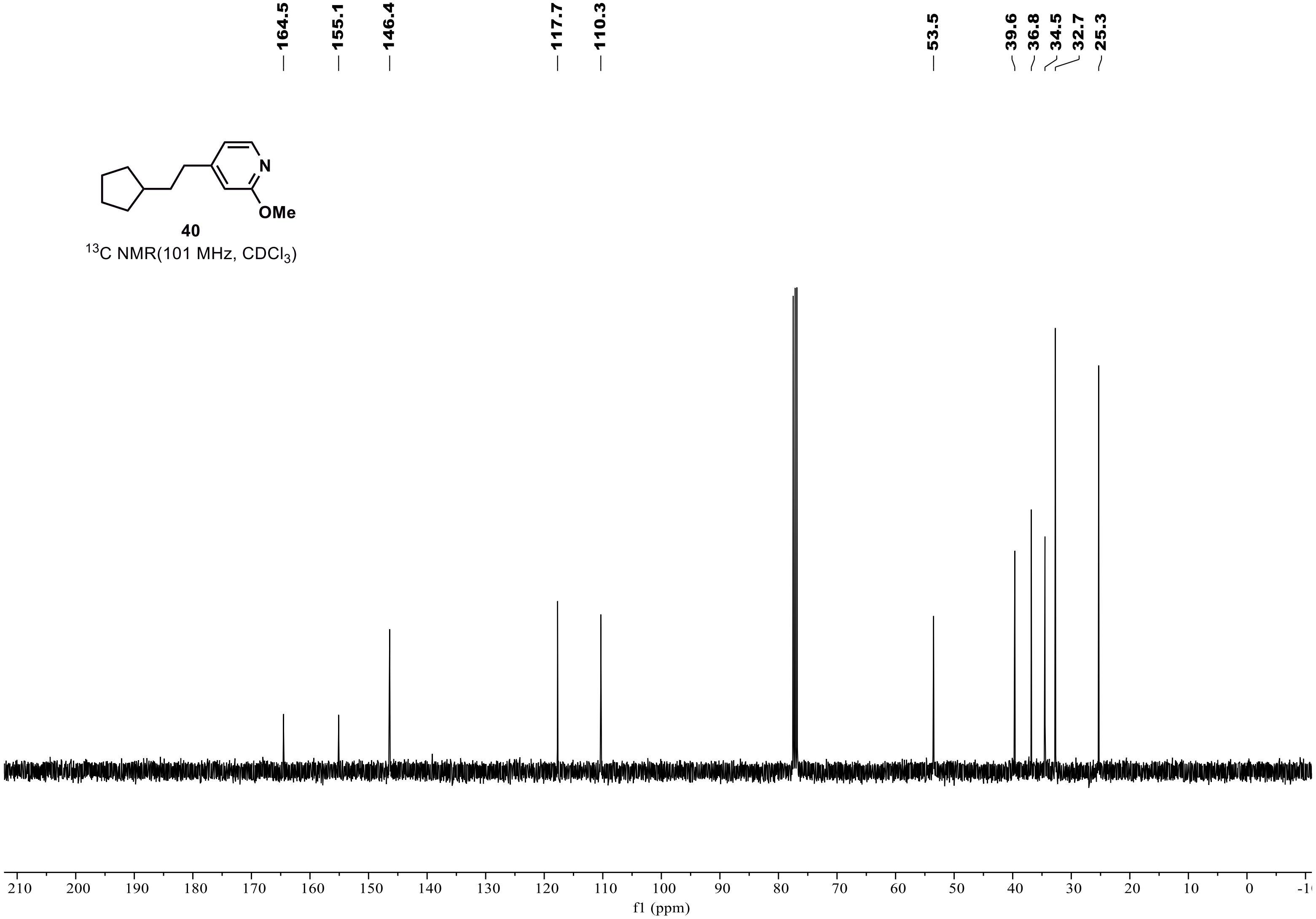




39

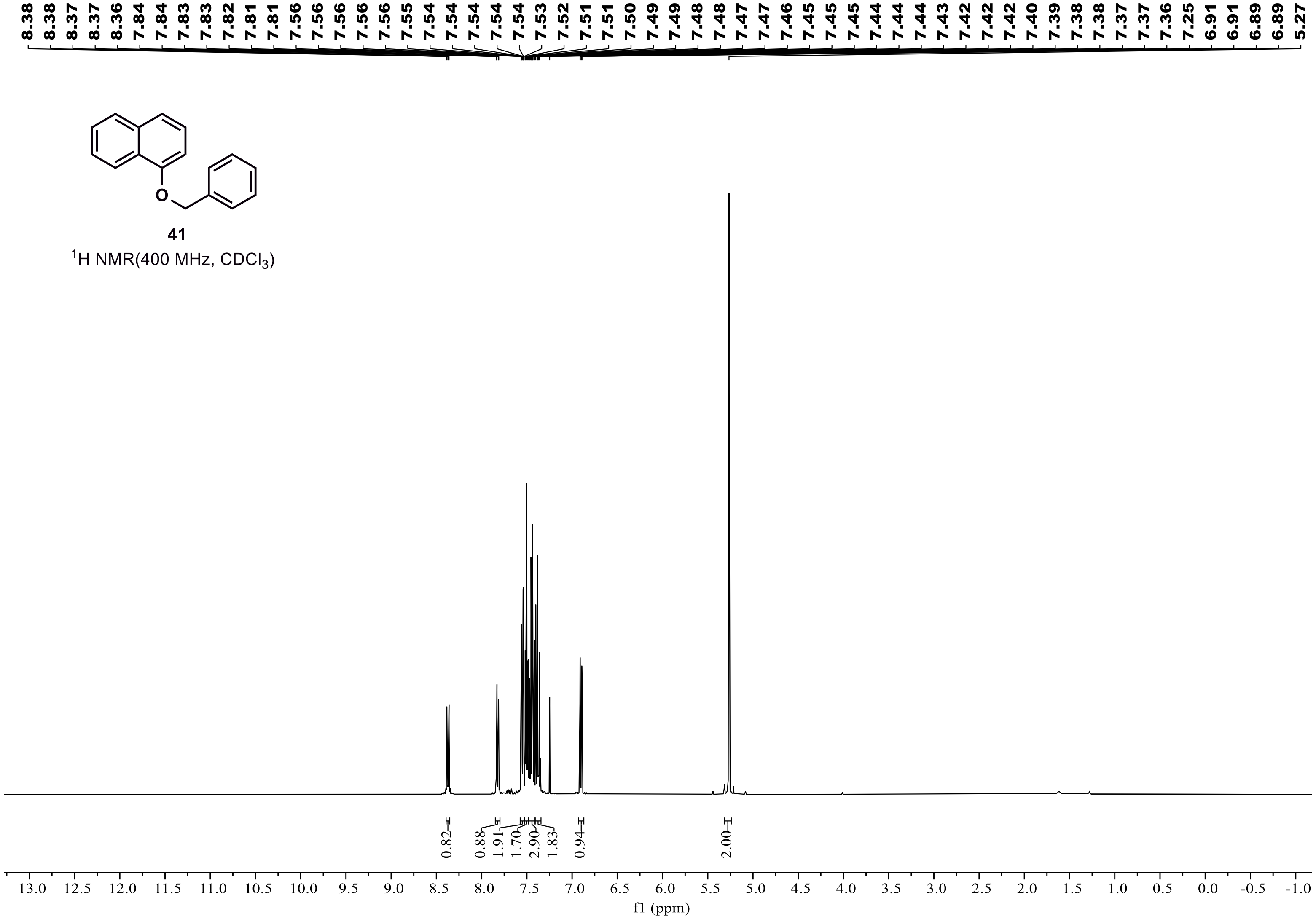
^{13}C NMR(101 MHz, $CDCl_3$)


8.04
8.03
7.26
6.71
6.71
6.70
6.70
6.56
6.56
6.56
6.55
3.92
2.57
2.56
2.55
2.55
2.55
2.53
1.78
1.78
1.77
1.77
1.77
1.77
1.76
1.76
1.75
1.75
1.75
1.75
1.63
1.62
1.62
1.61
1.61
1.61
1.61
1.60
1.60
1.59
1.59
1.59
1.59
1.58
1.58
1.57
1.57
1.52
1.51
1.51
1.50
1.50
1.49
1.11
1.10
1.10
1.10
N
OMe
40
^{1}H NMR(400 MHz, CDCl$_3$)
0.87
0.91
0.84
3.00
2.03
3.16
4.24
1.98
2.07
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



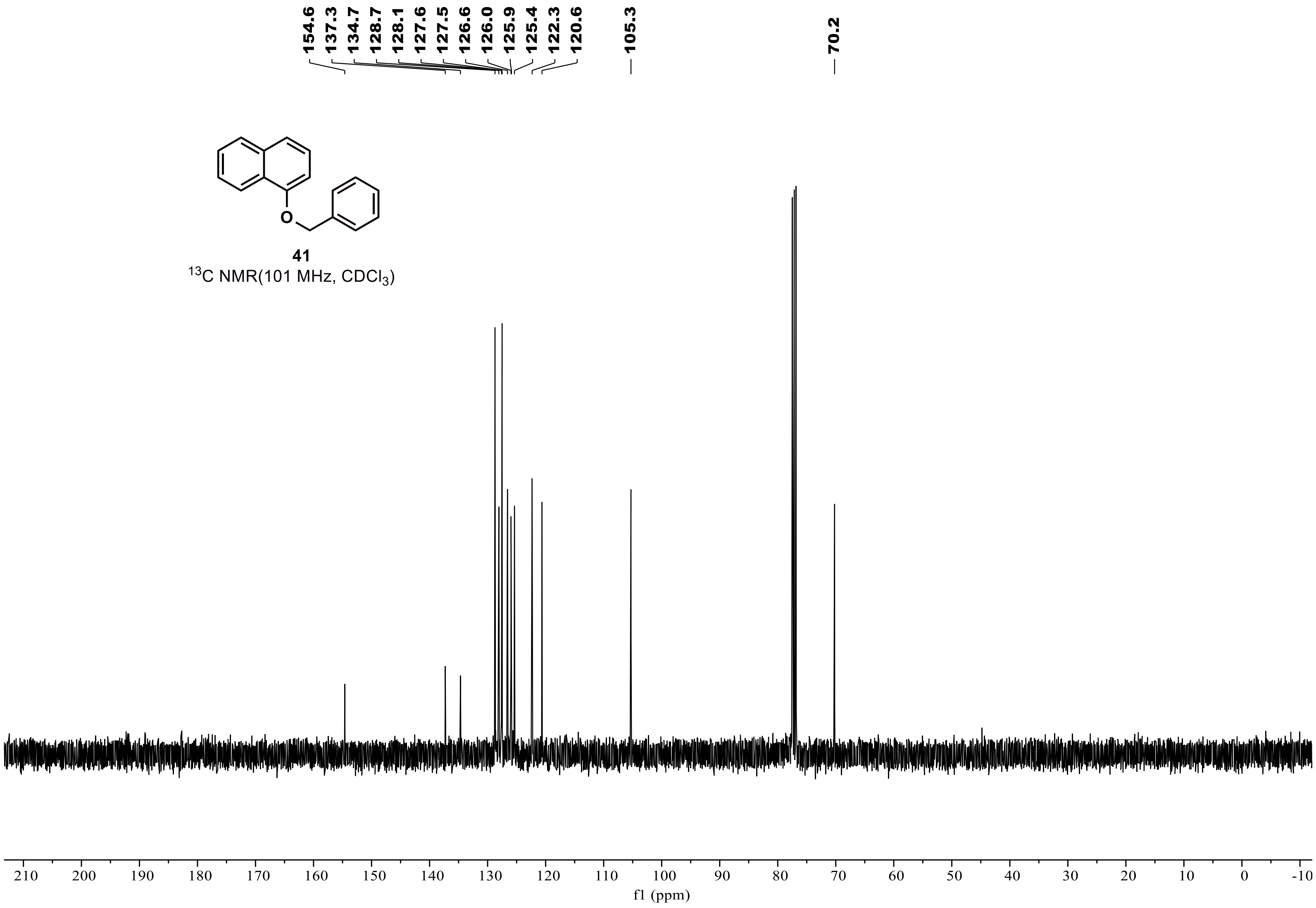
40

^{13}C NMR(101 MHz, CDCl$_3$)



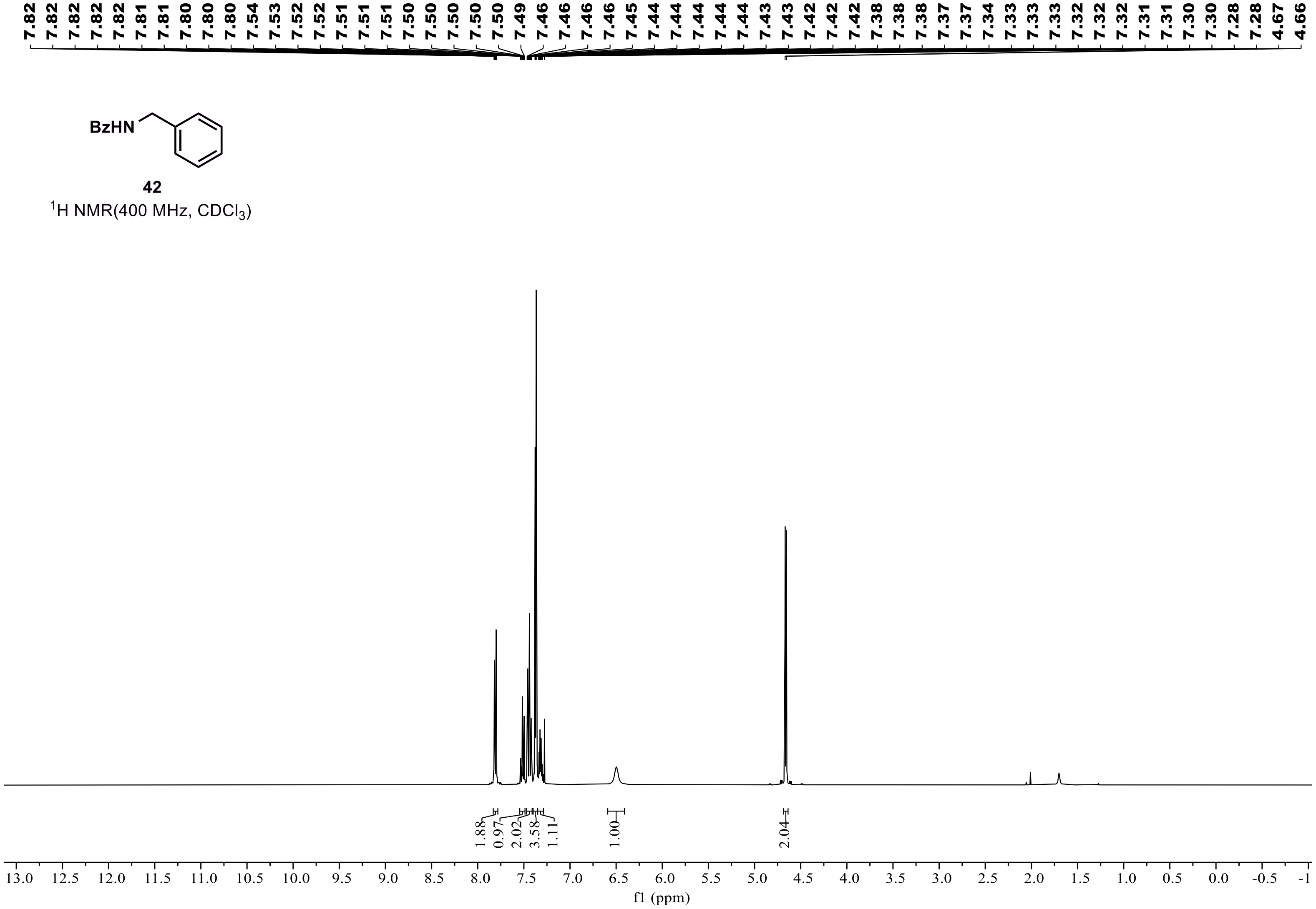
41

^{1}H NMR(400 MHz, $CDCl_3$)



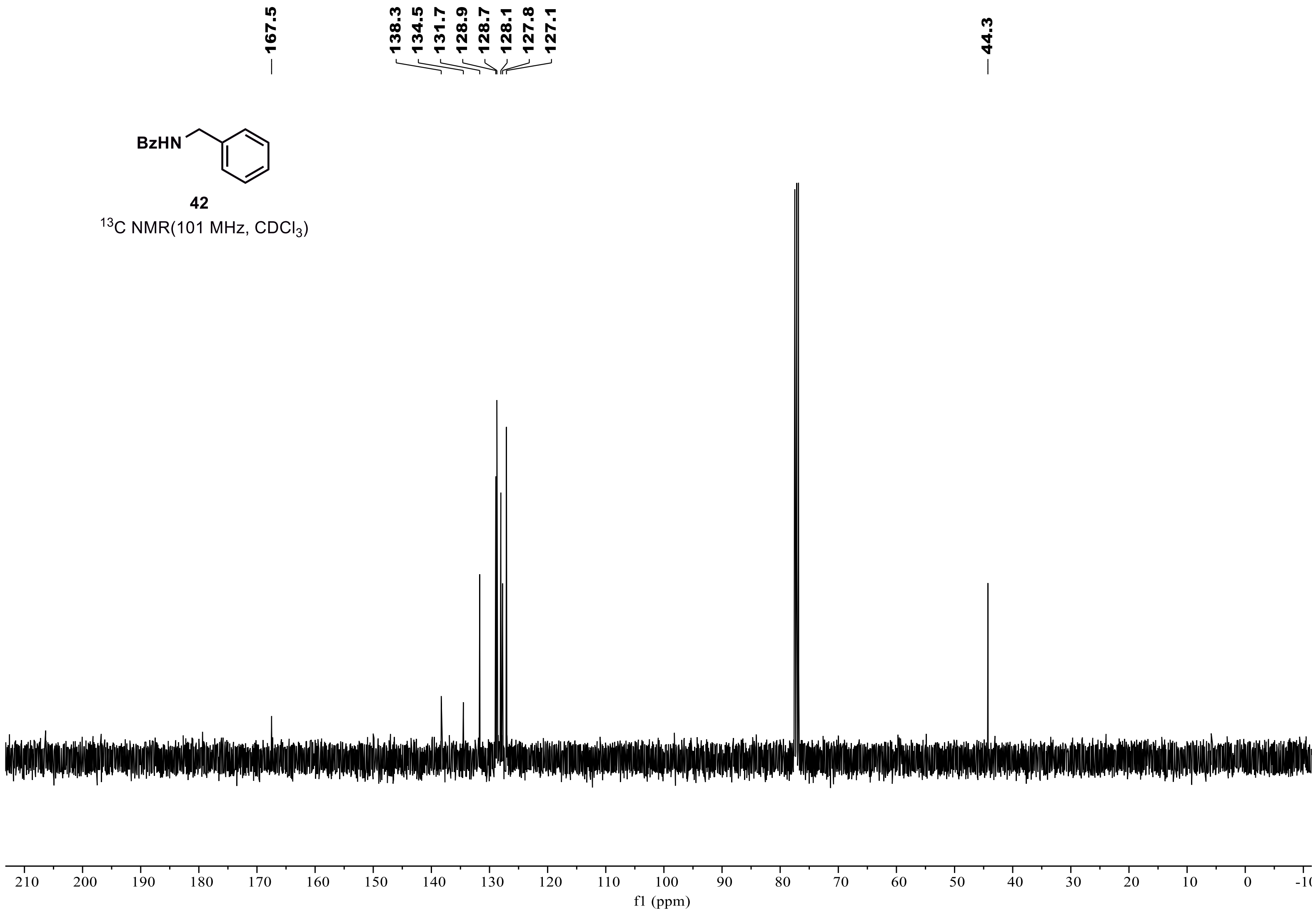
41

^{13}C NMR(101 MHz, $CDCl_3$)



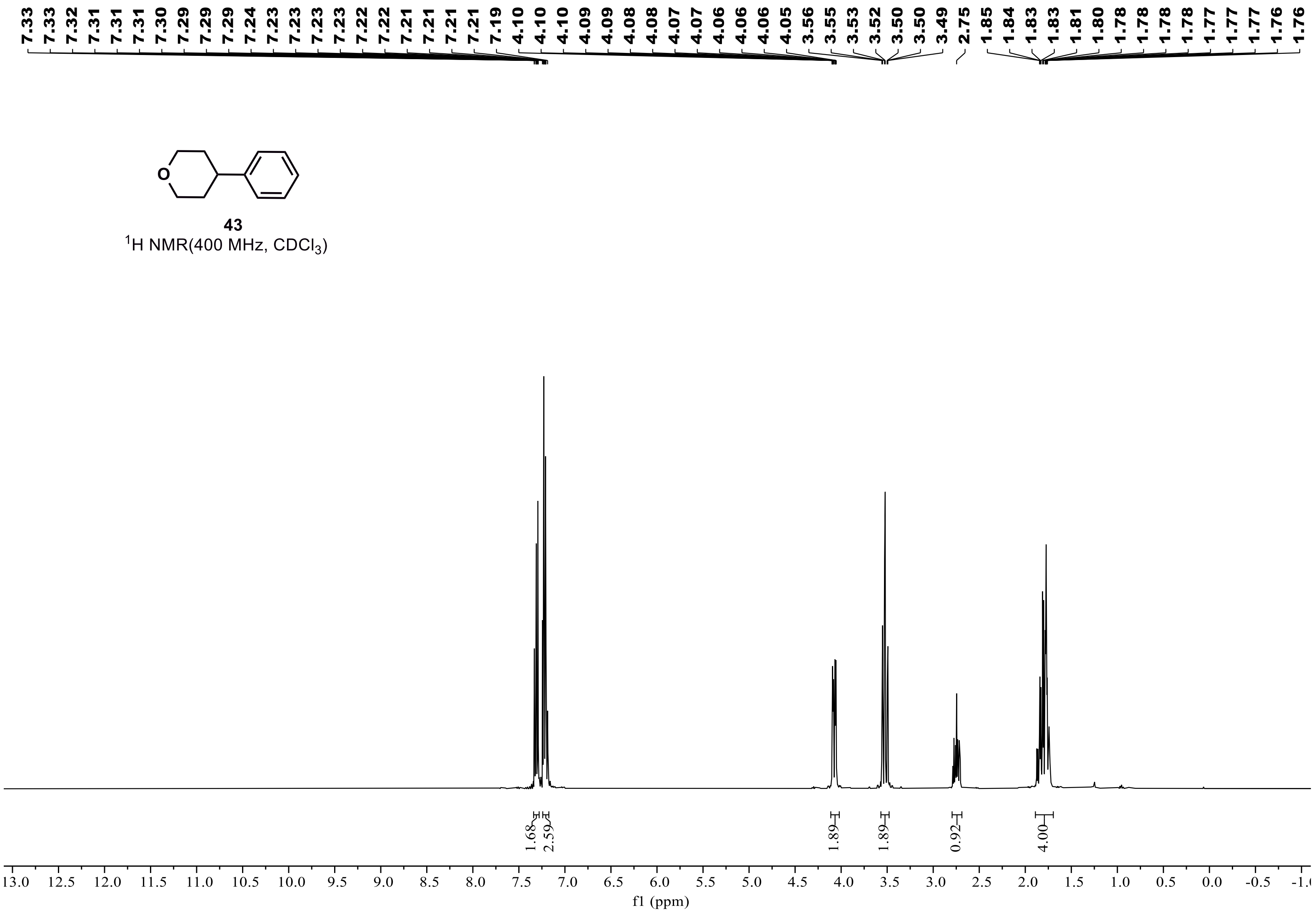
42

^{1}H NMR(400 MHz, $CDCl_3$)



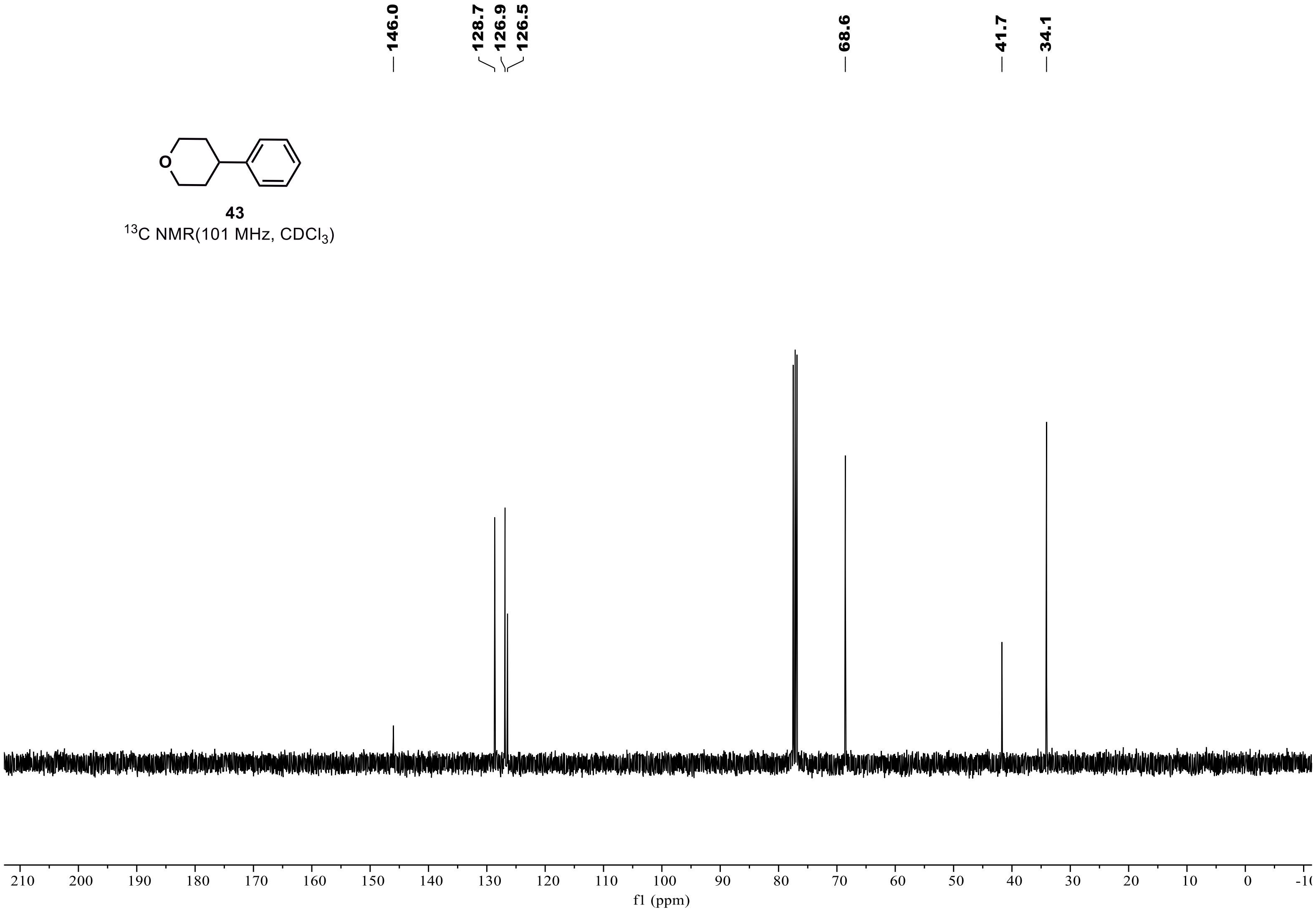
42

^{13}C NMR(101 MHz, $CDCl_3$)



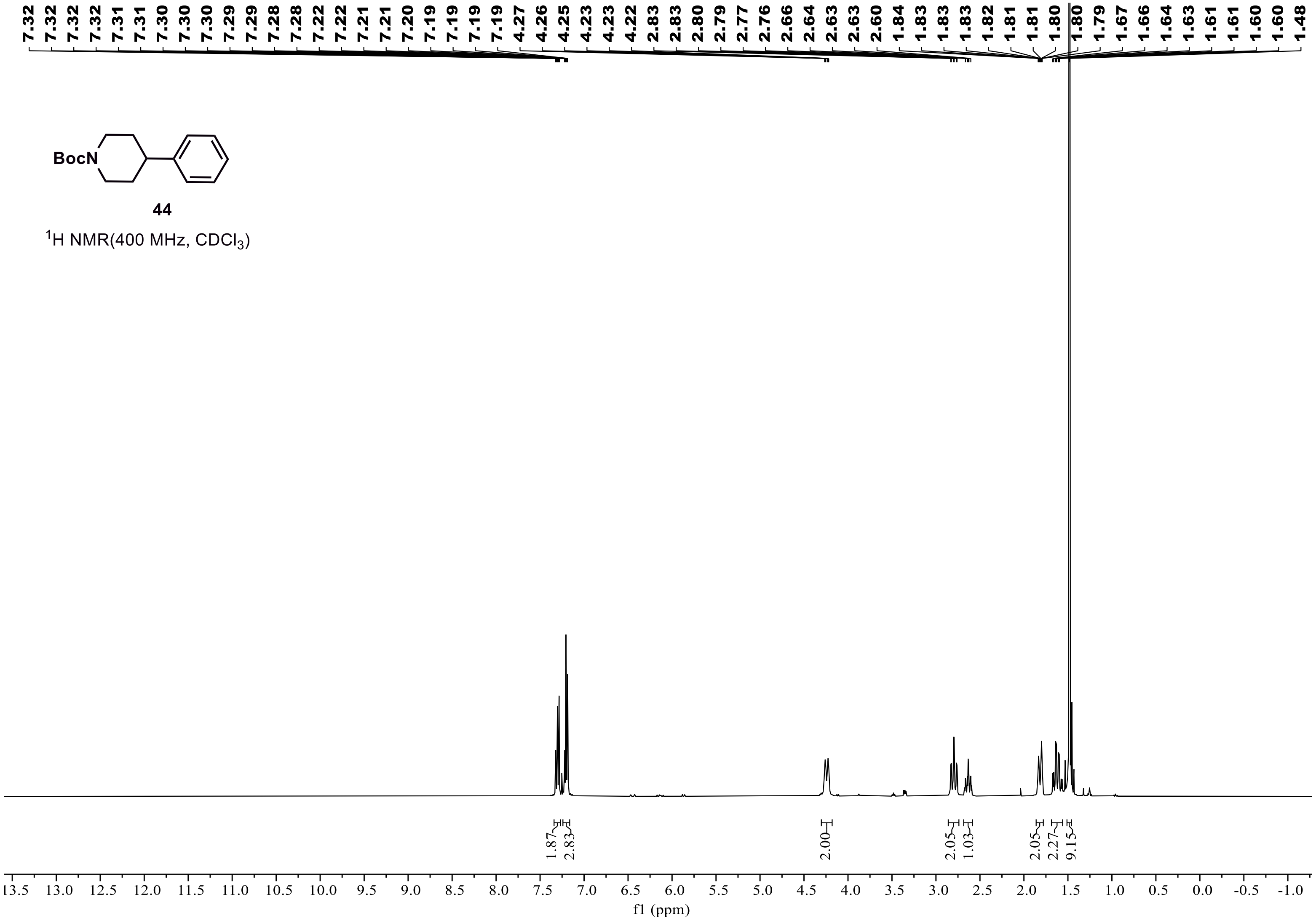
43

^{1}H NMR(400 MHz, $CDCl_3$)



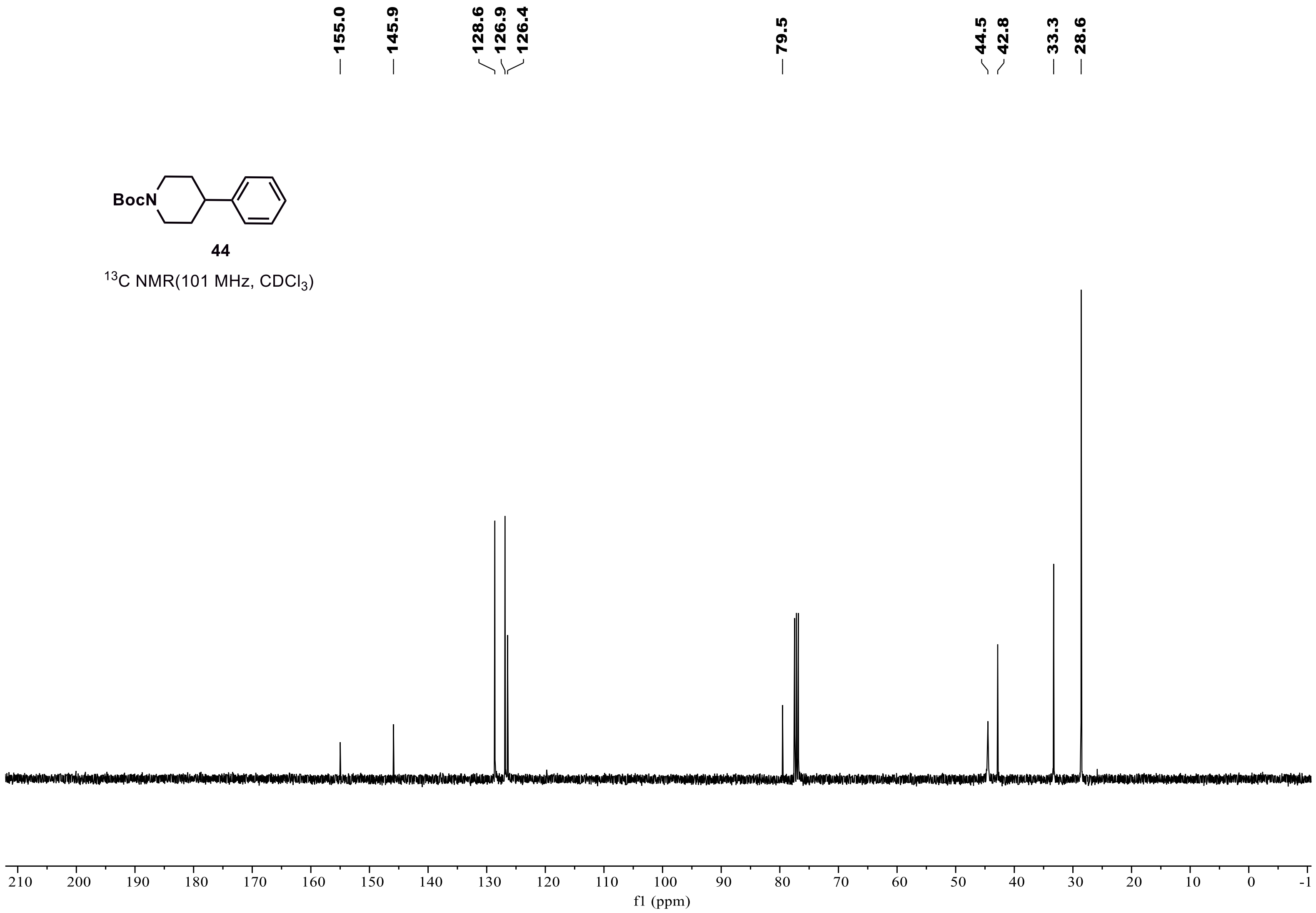
43

^{13}C NMR(101 MHz, $CDCl_3$)



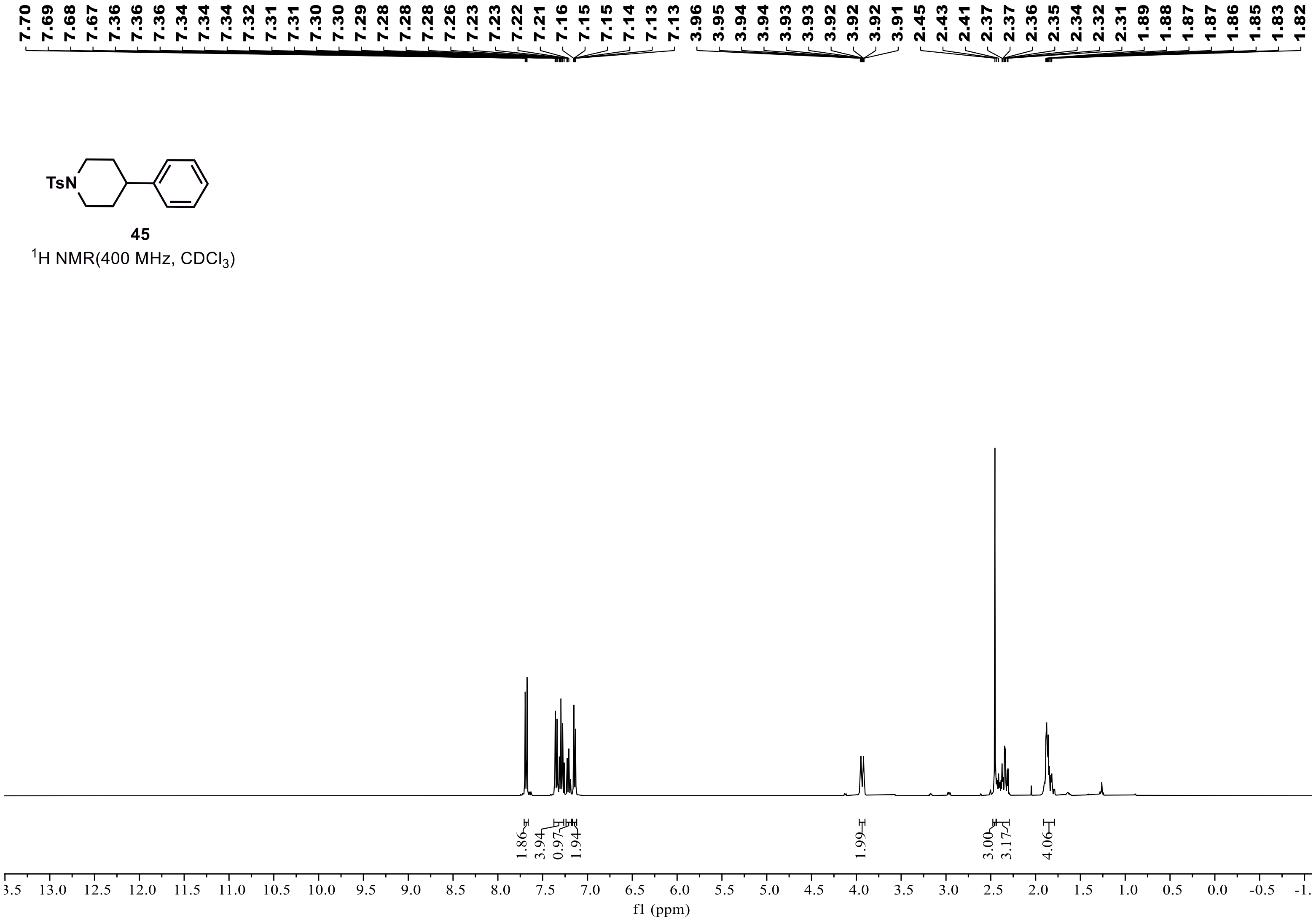
44

^{1}H NMR(400 MHz, $CDCl_3$)



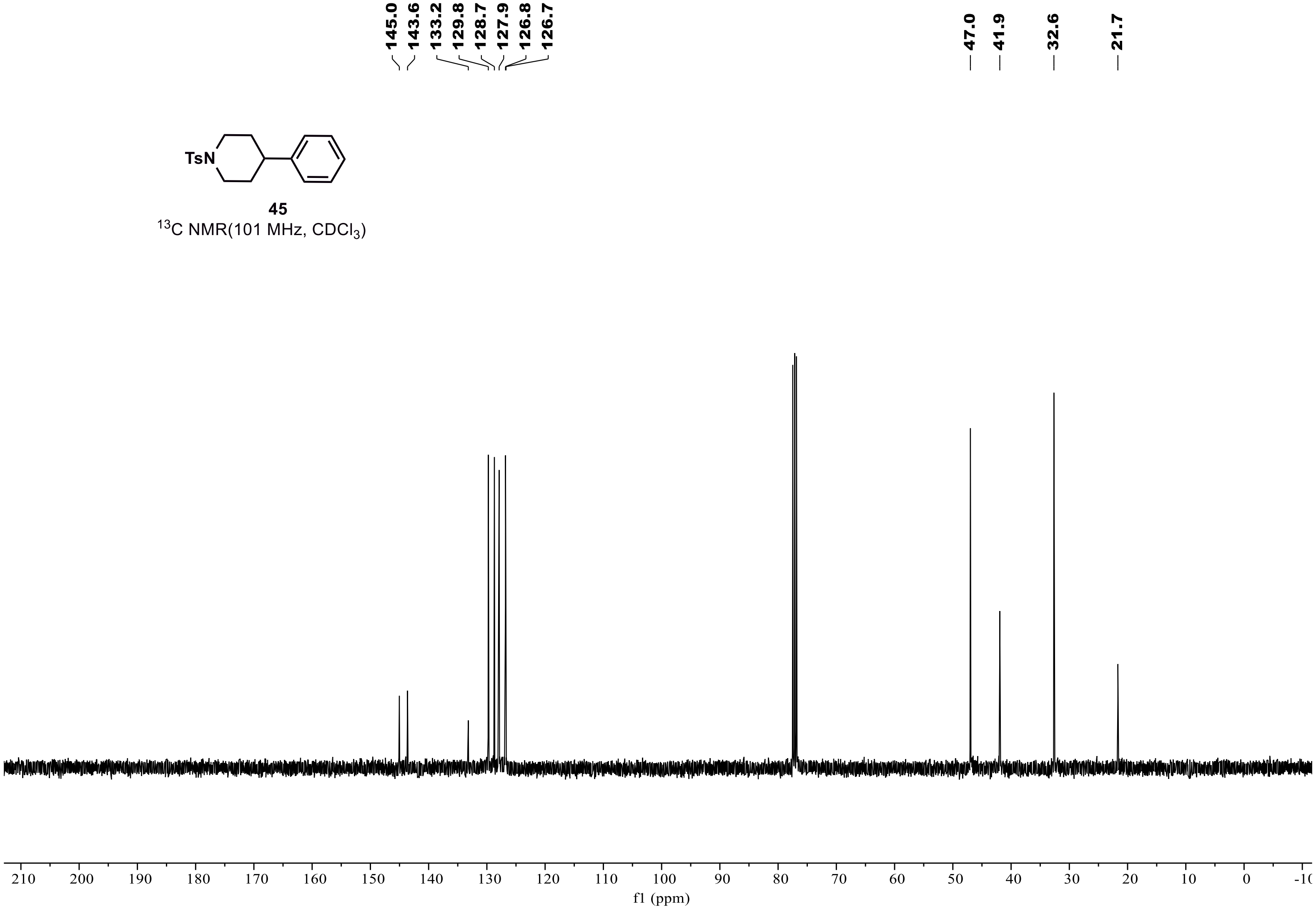
44

^{13}C NMR(101 MHz, CDCl$_3$)



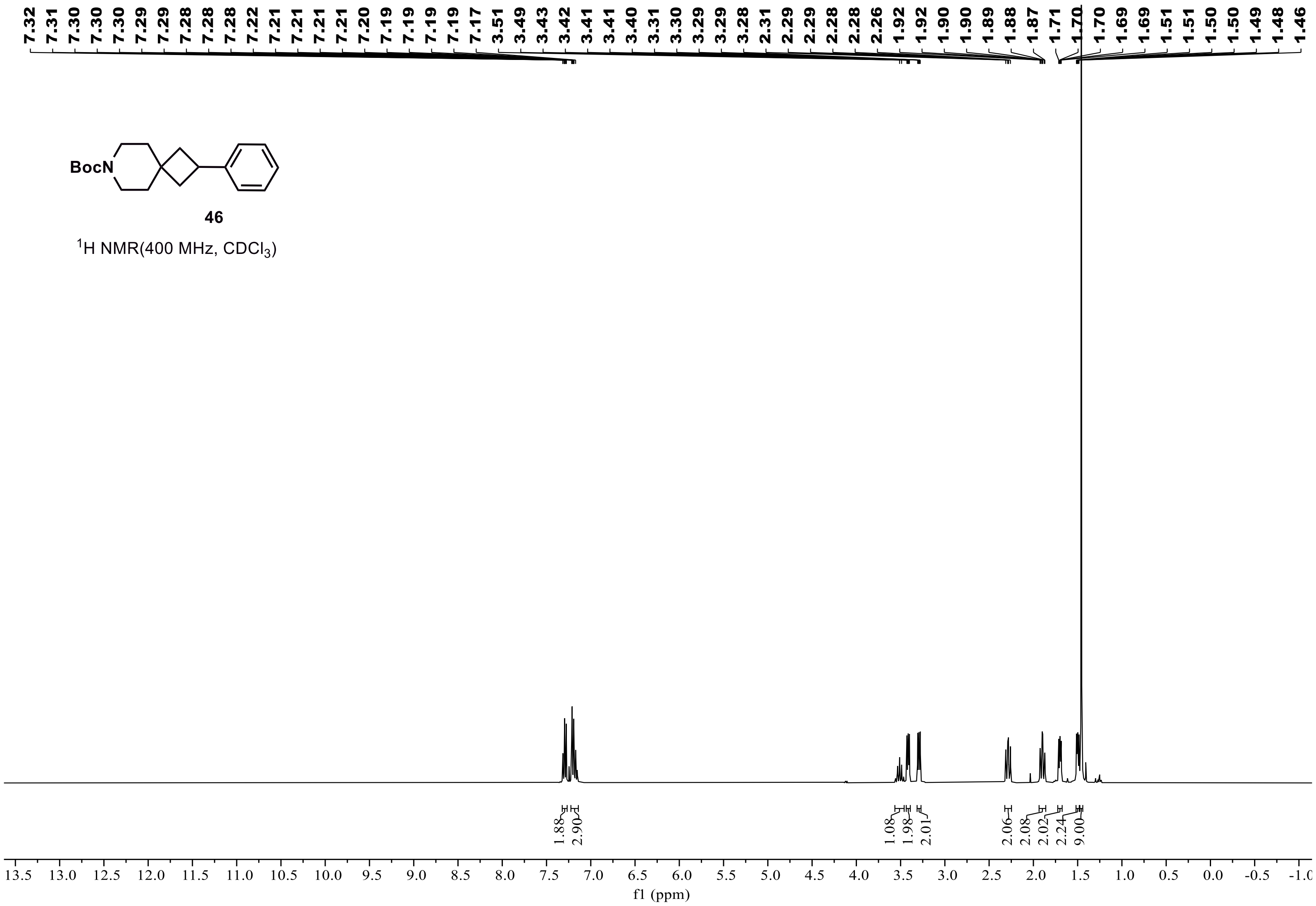
45

^{1}H NMR(400 MHz, CDCl$_3$)



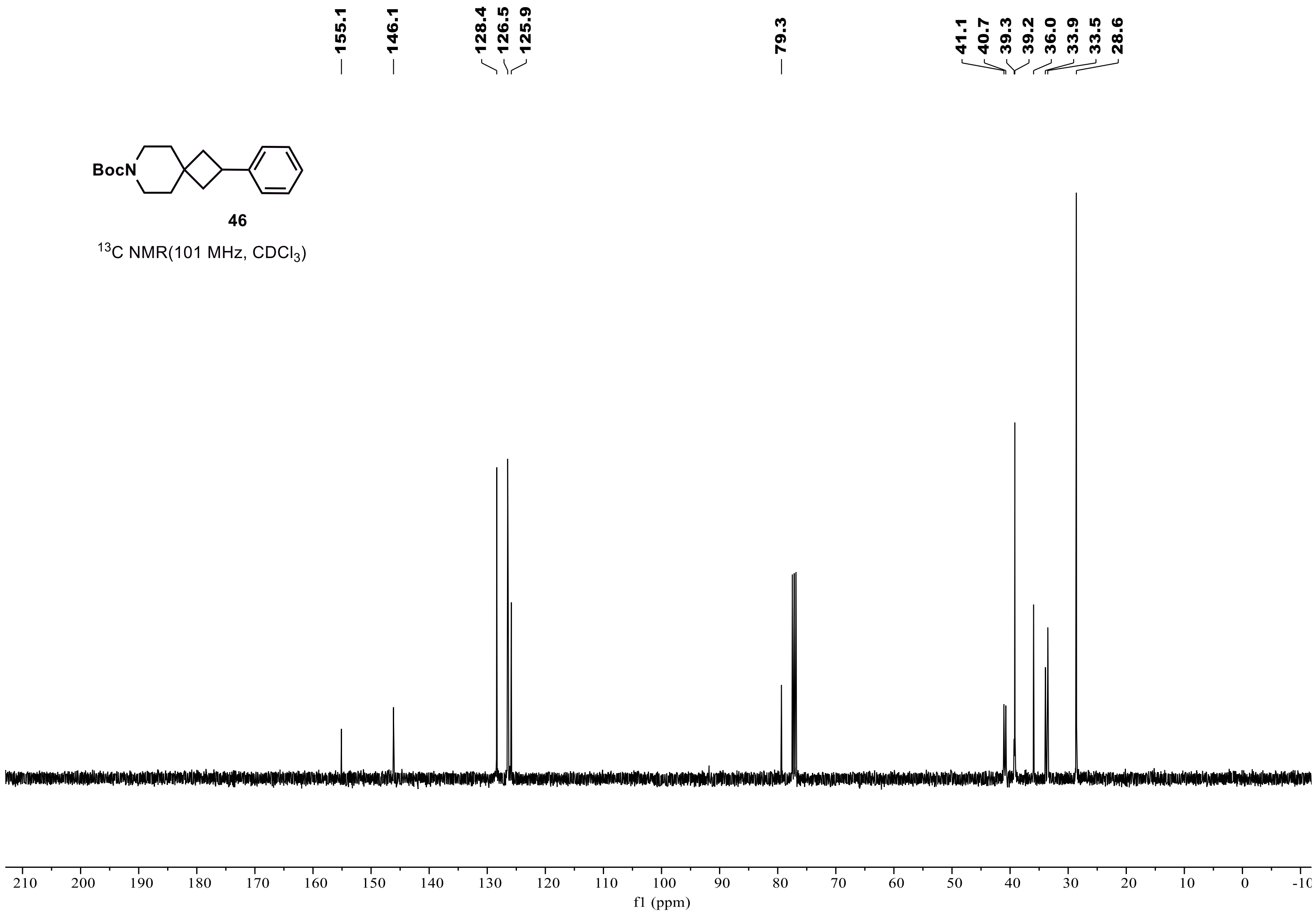
45

^{13}C NMR(101 MHz, $CDCl_3$)



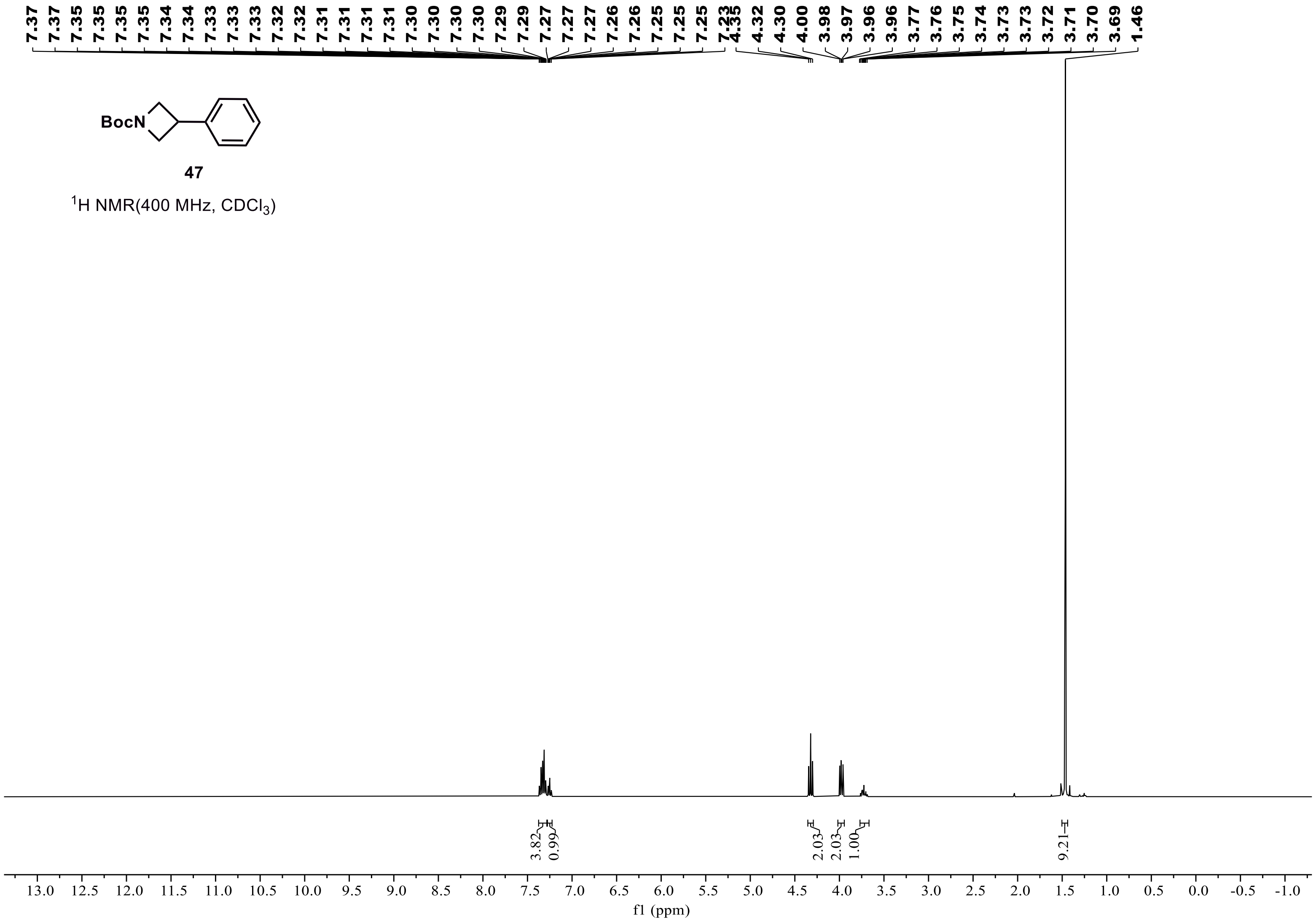
46

^{1}H NMR(400 MHz, CDCl$_3$)



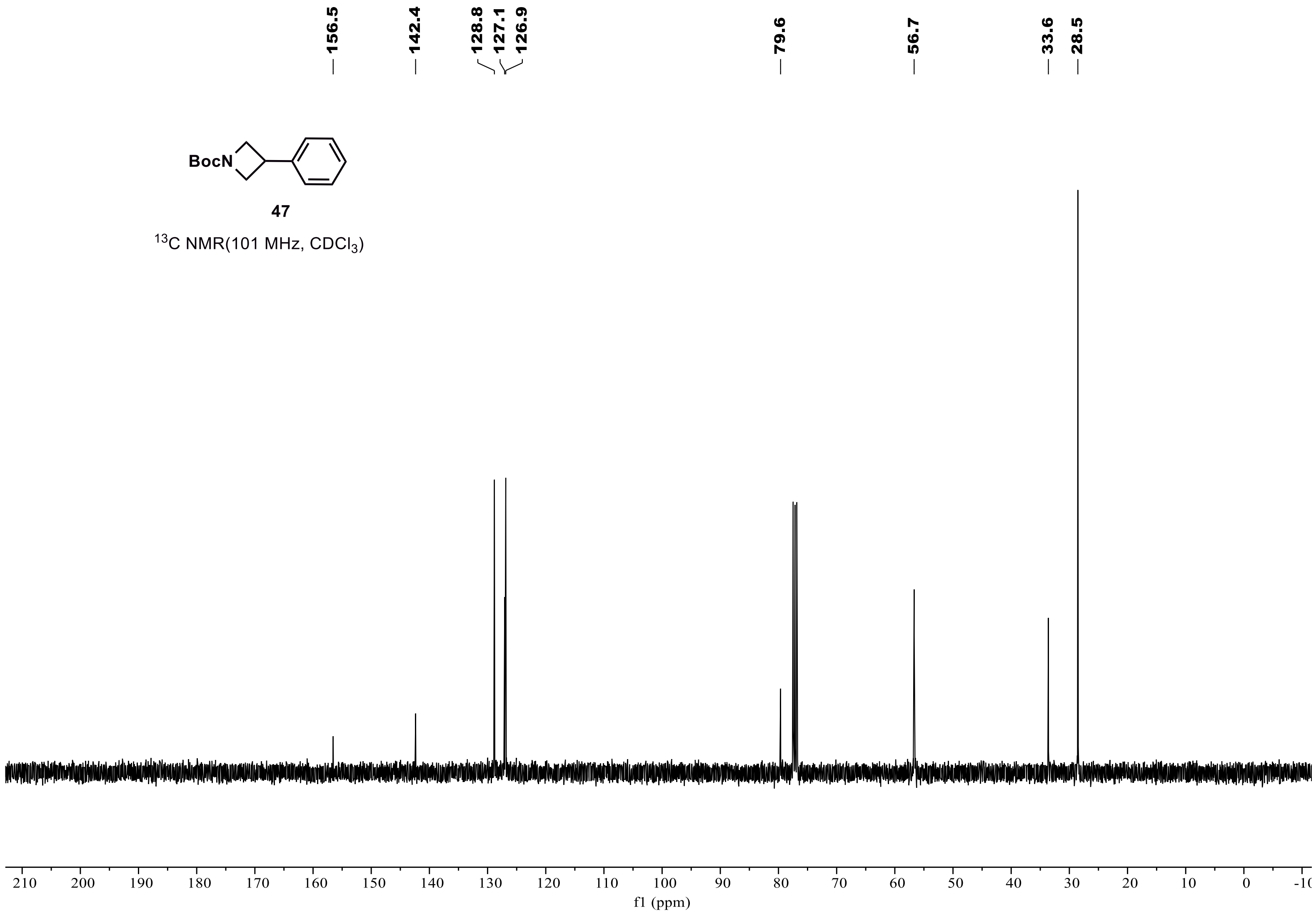
46

^{13}C NMR(101 MHz, $CDCl_3$)



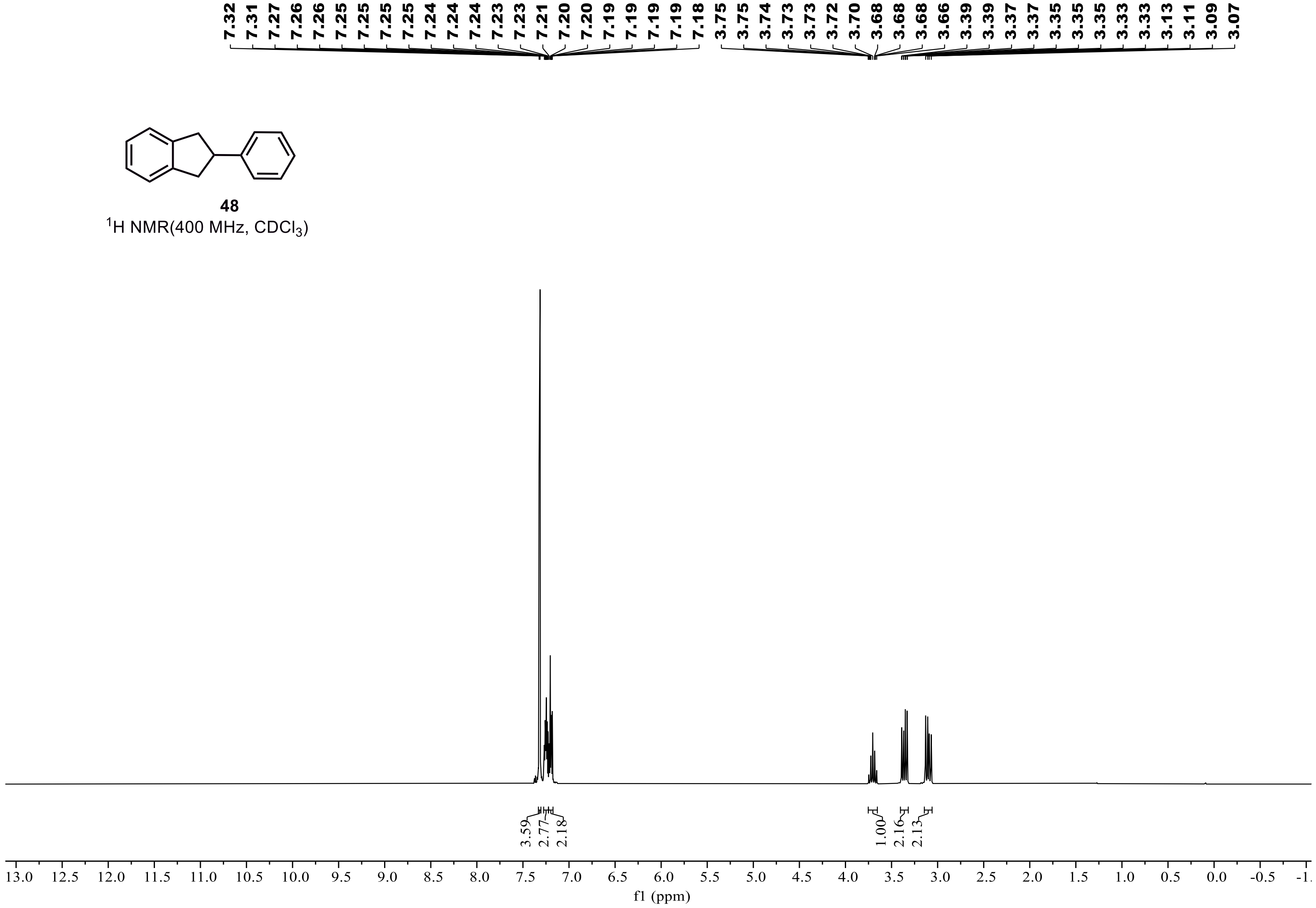
47

^{1}H NMR(400 MHz, $CDCl_3$)



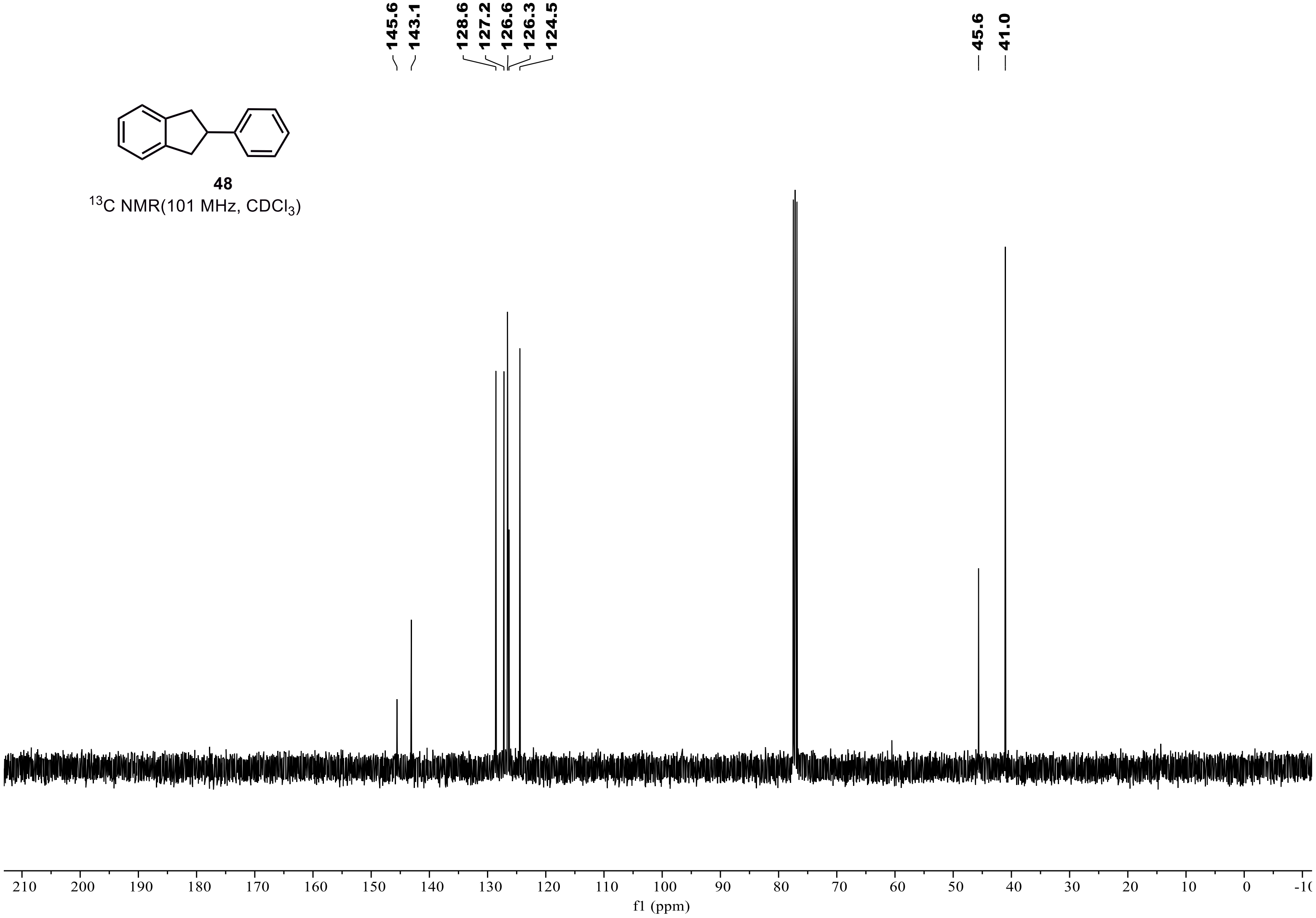
47

^{13}C NMR(101 MHz, CDCl$_3$)

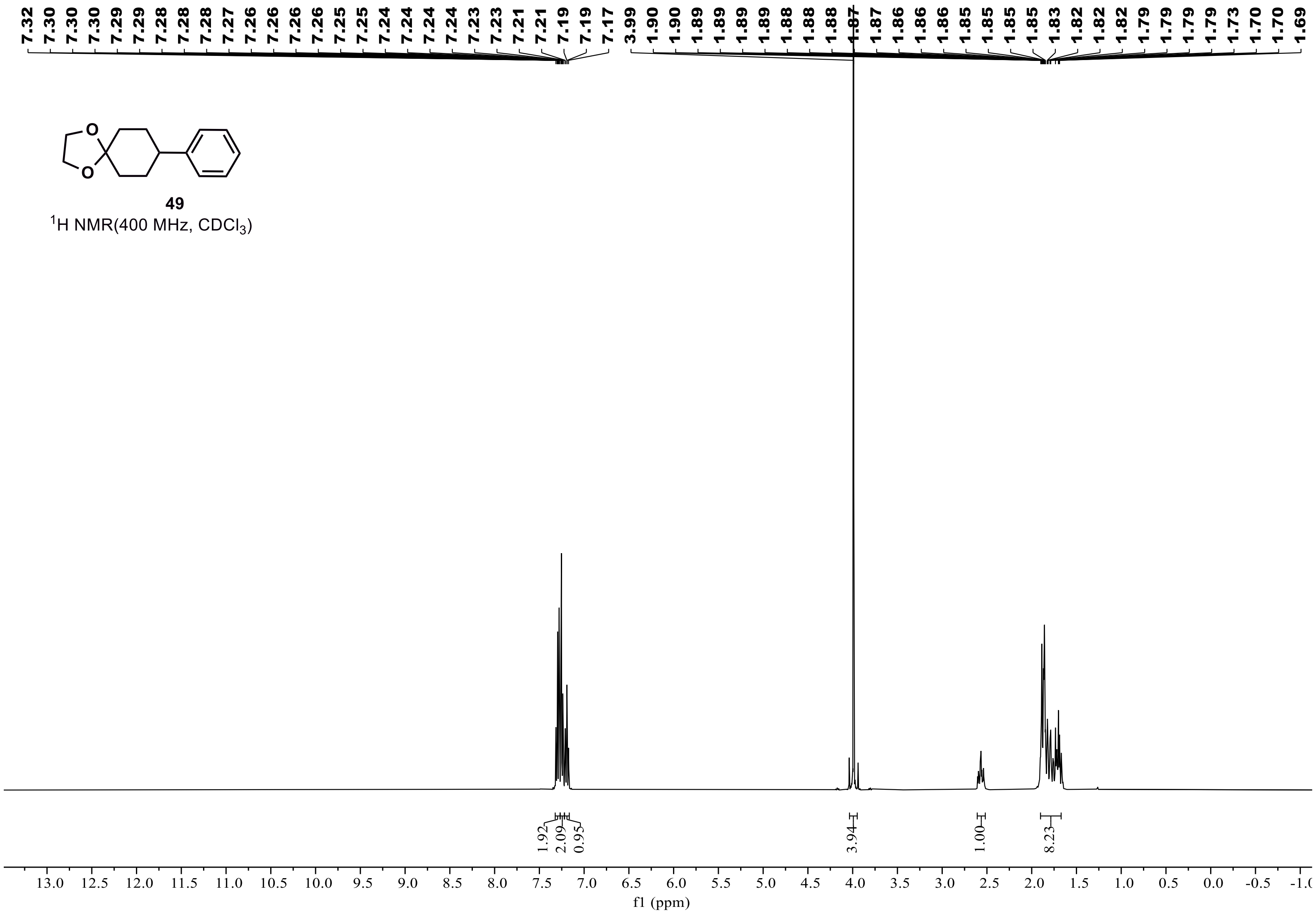


48

^1H NMR(400 MHz, $CDCl_3$)

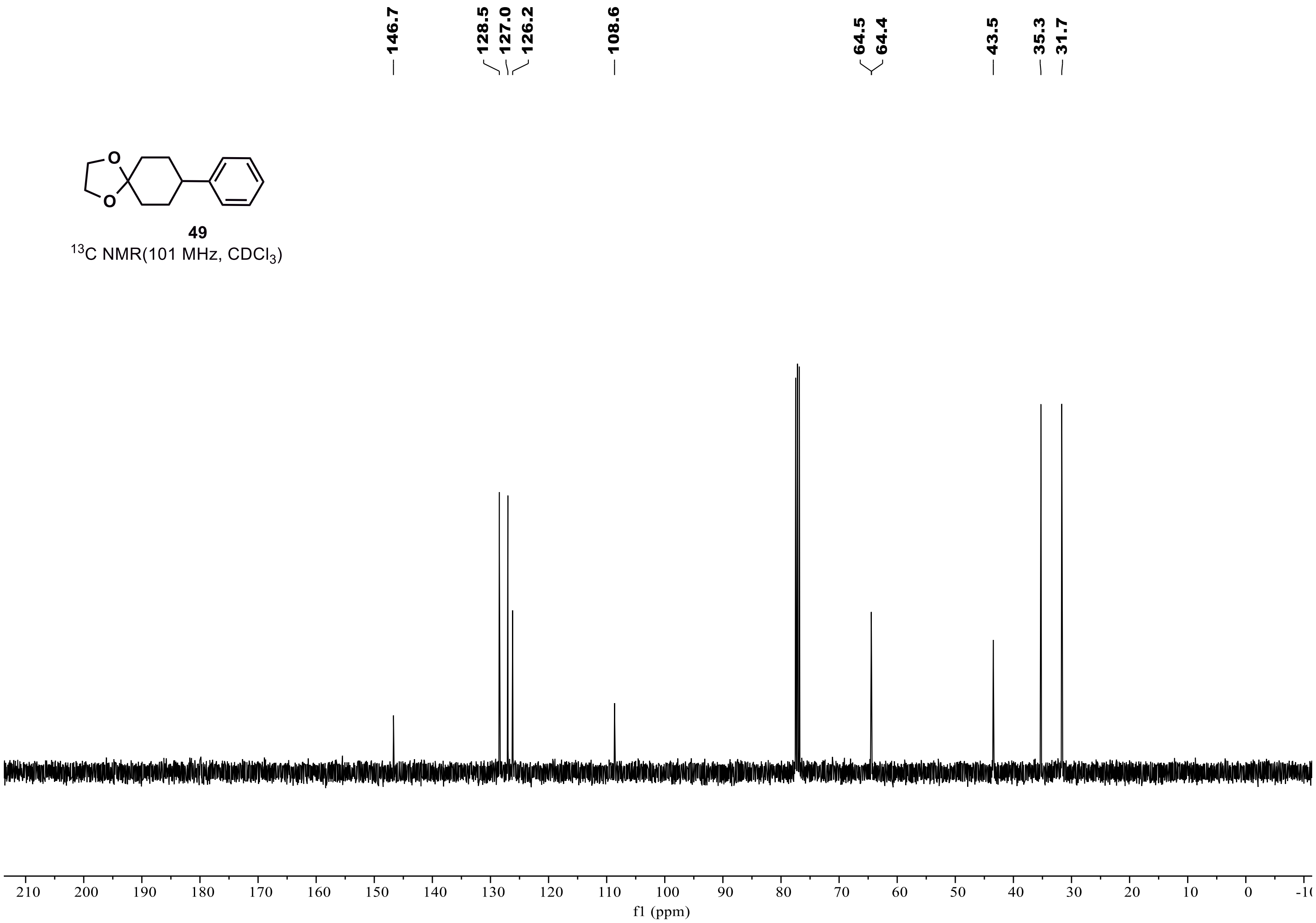

48

^{13}C NMR(101 MHz, CDCl$_3$)



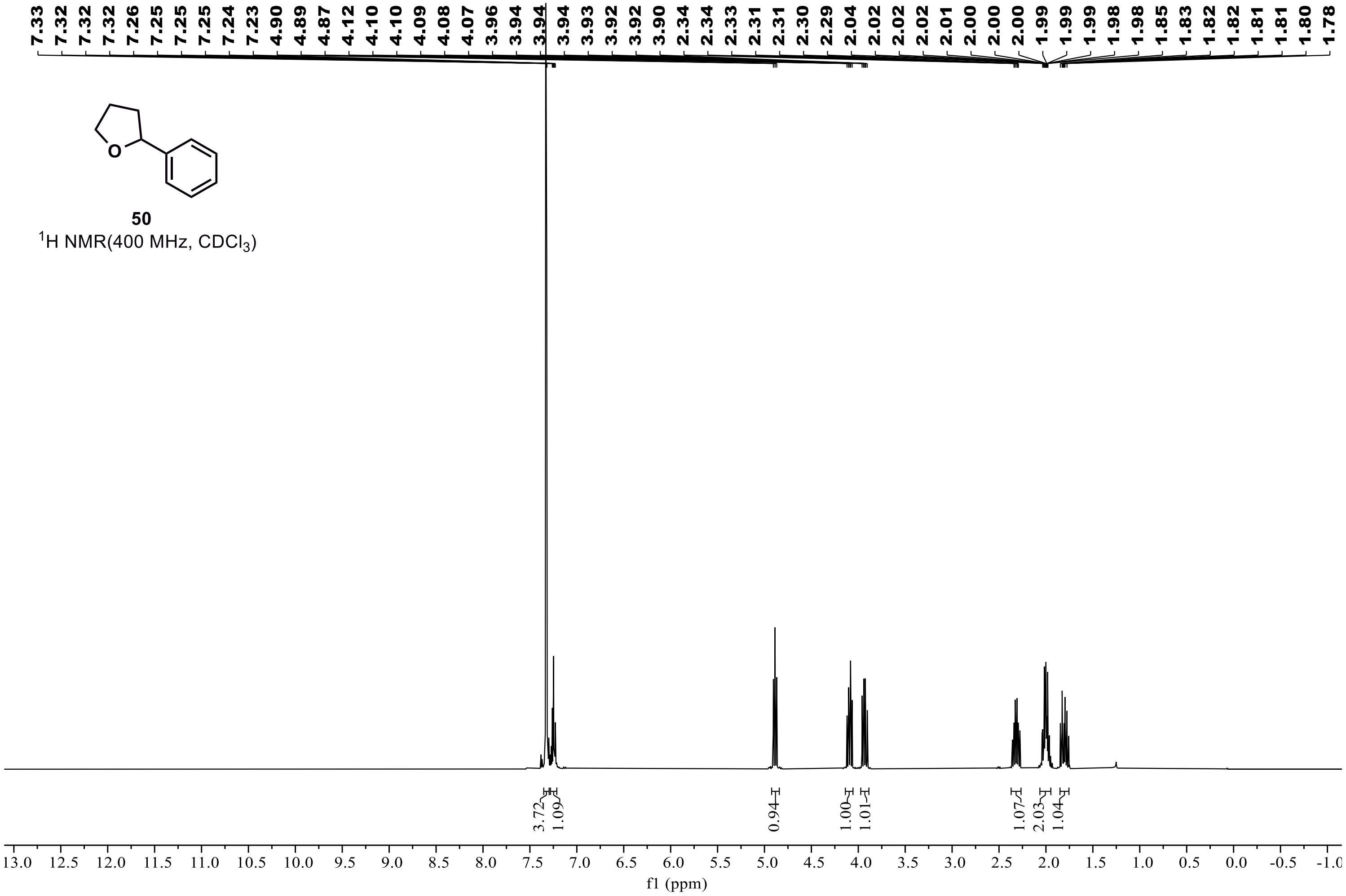
49

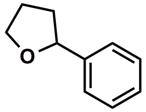
^{1}H NMR(400 MHz, $CDCl_3$)



49

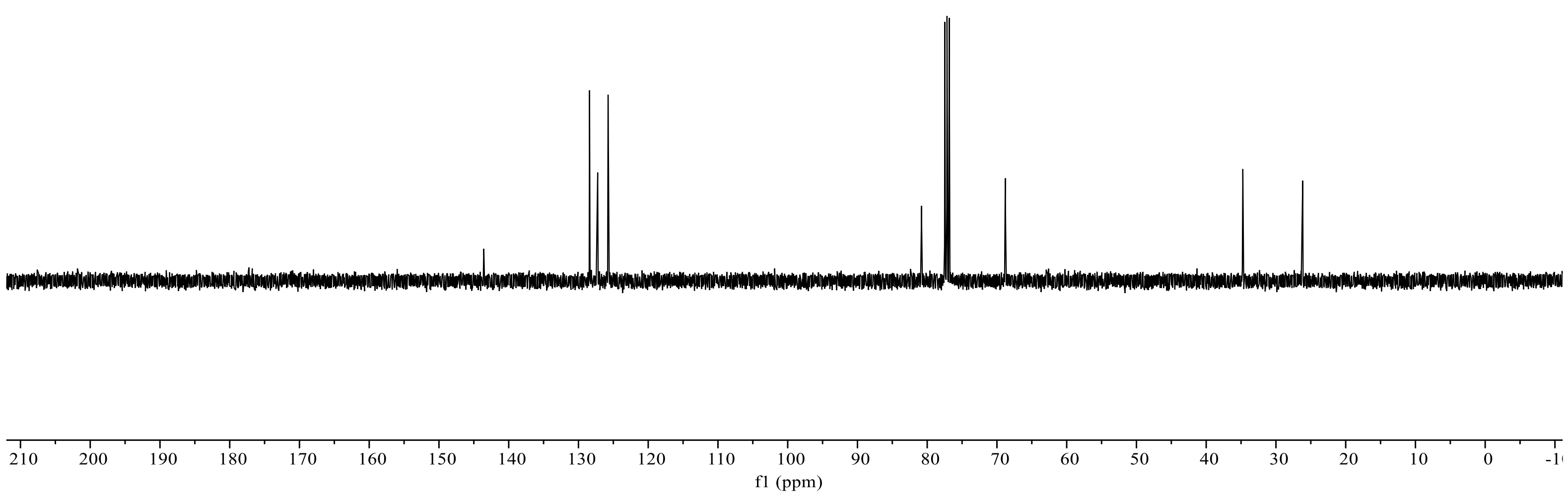
^{13}C NMR(101 MHz, CDCl$_3$)

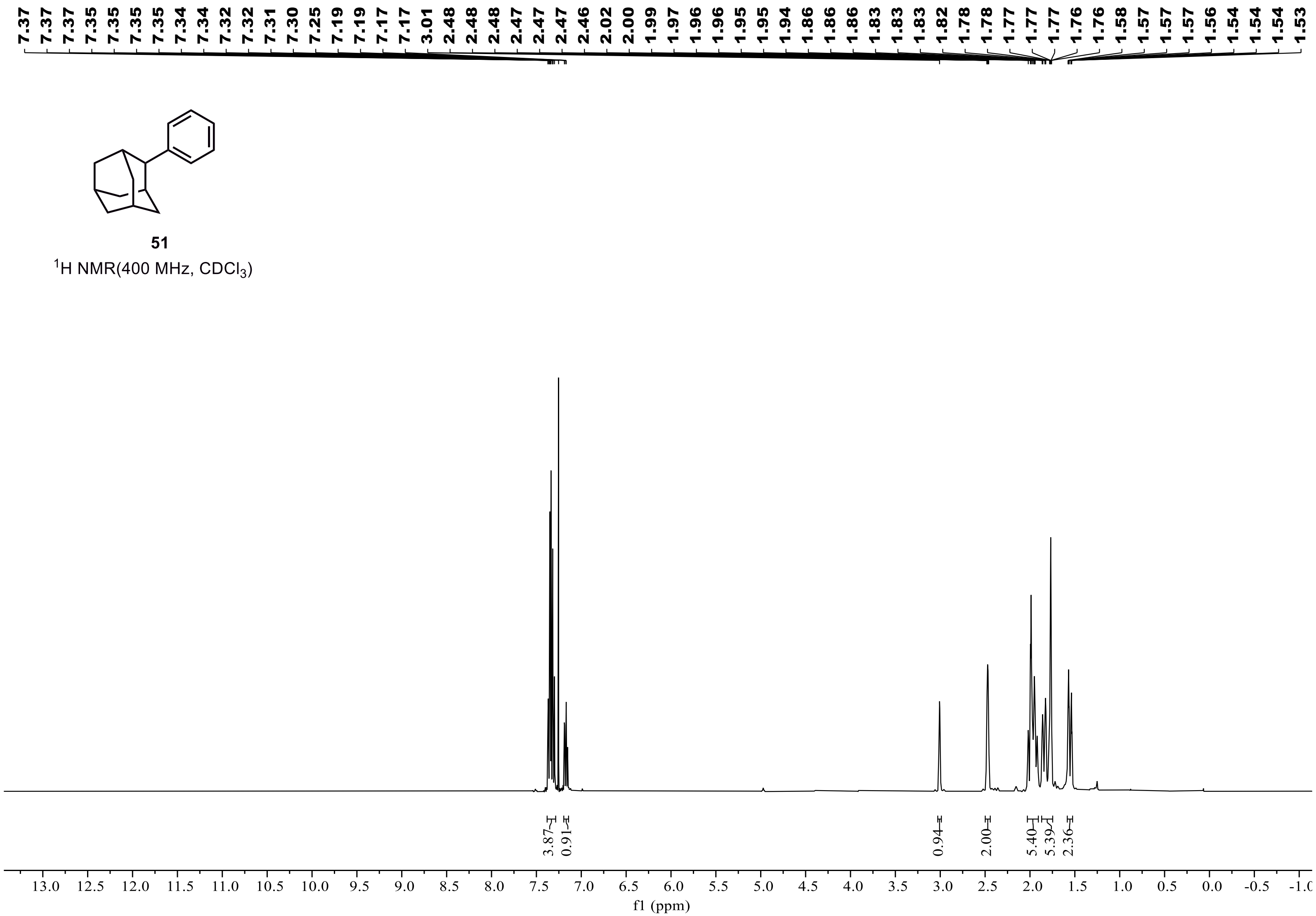

7.33
7.32
7.32
7.32
7.26
7.25
7.25
7.25
7.24
7.23
4.90
4.89
4.87
4.12
4.10
4.10
4.09
4.08
4.07
3.96
3.94
3.94
3.94
3.93
3.92
3.92
3.90
2.34
2.34
2.33
2.31
2.31
2.30
2.29
2.04
2.02
2.02
2.02
2.01
2.00
2.00
1.99
1.99
1.99
1.98
1.98
1.85
1.83
1.82
1.82
1.81
1.81
1.80
1.78
O
50
^{1}H NMR(400 MHz, CDCl$_3$)
3.72
1.09
0.94
1.00
1.01
1.07
2.03
1.04
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



50

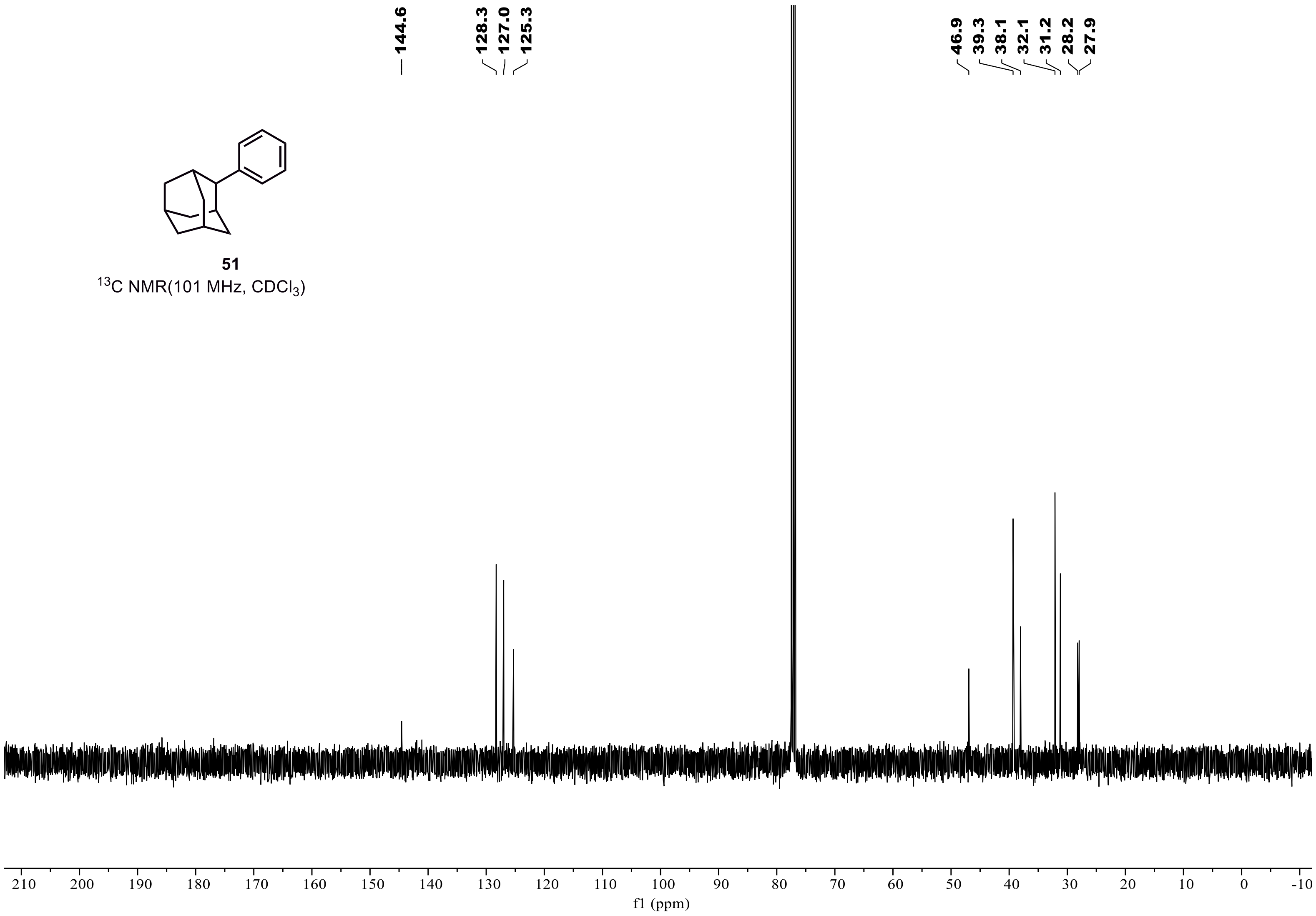
^{13}C NMR(101 MHz, CDCl$_3$)





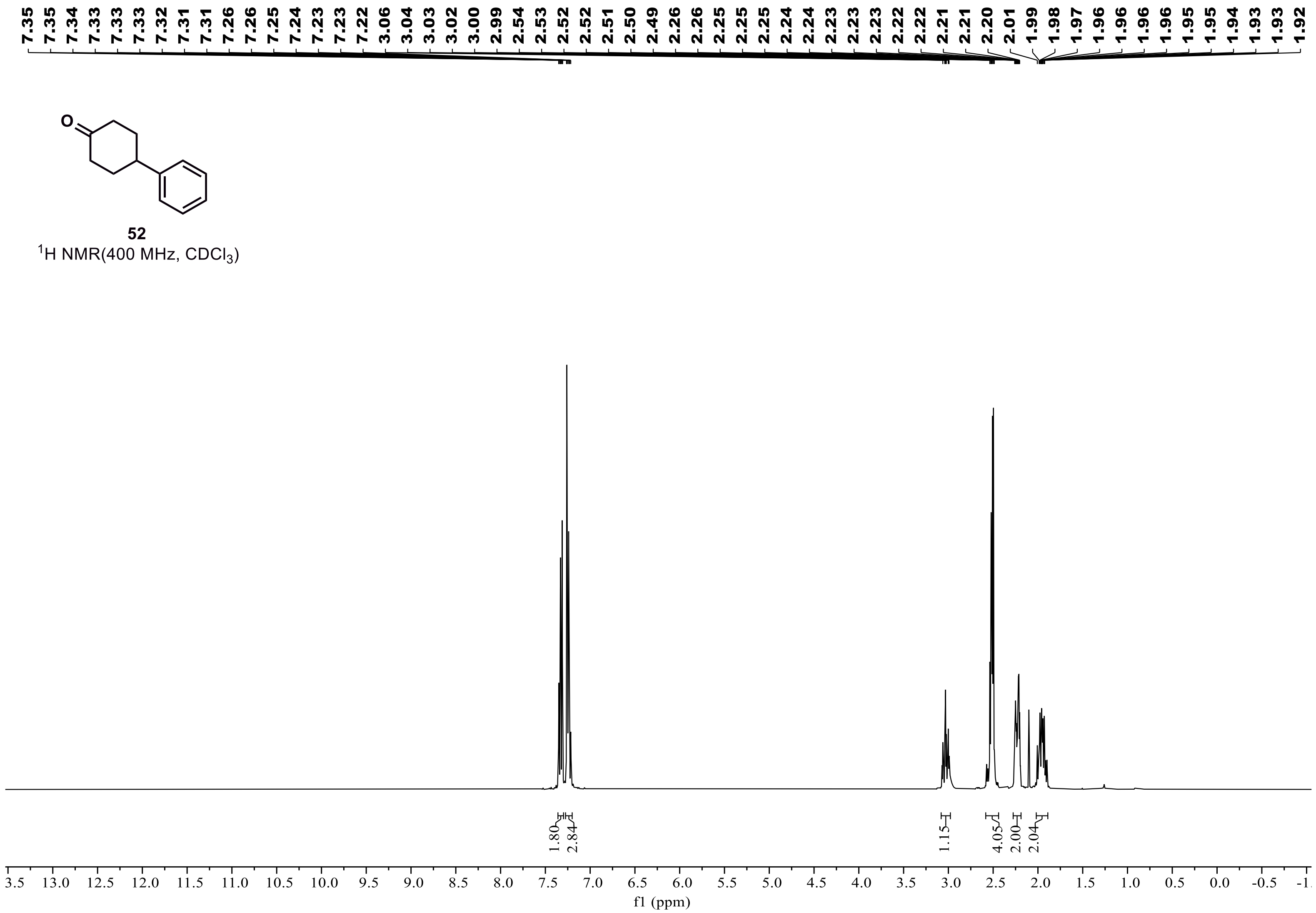
51

^{1}H NMR(400 MHz, $CDCl_3$)



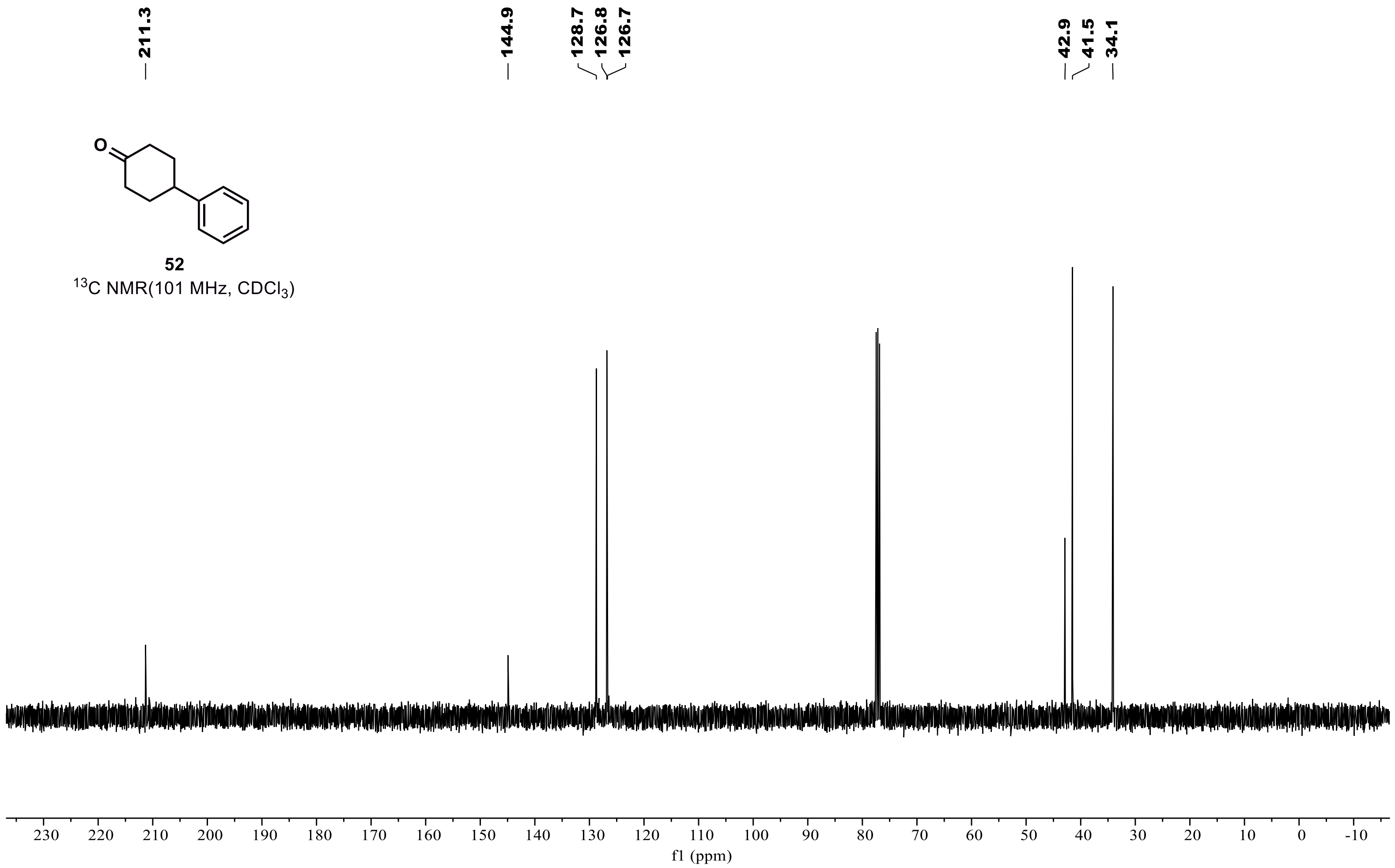
51

^{13}C NMR(101 MHz, CDCl$_3$)



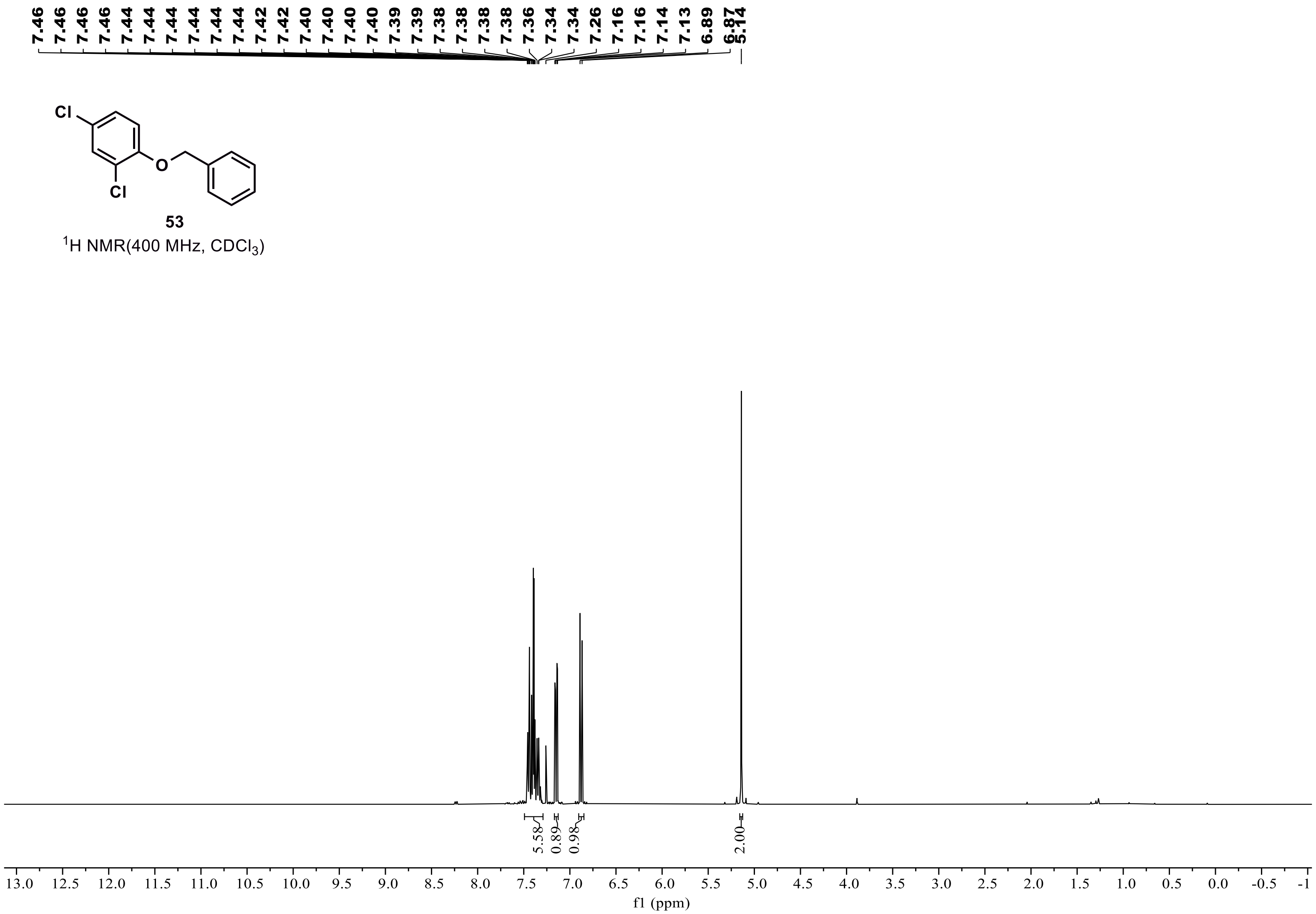
52

^{1}H NMR(400 MHz, $CDCl_3$)



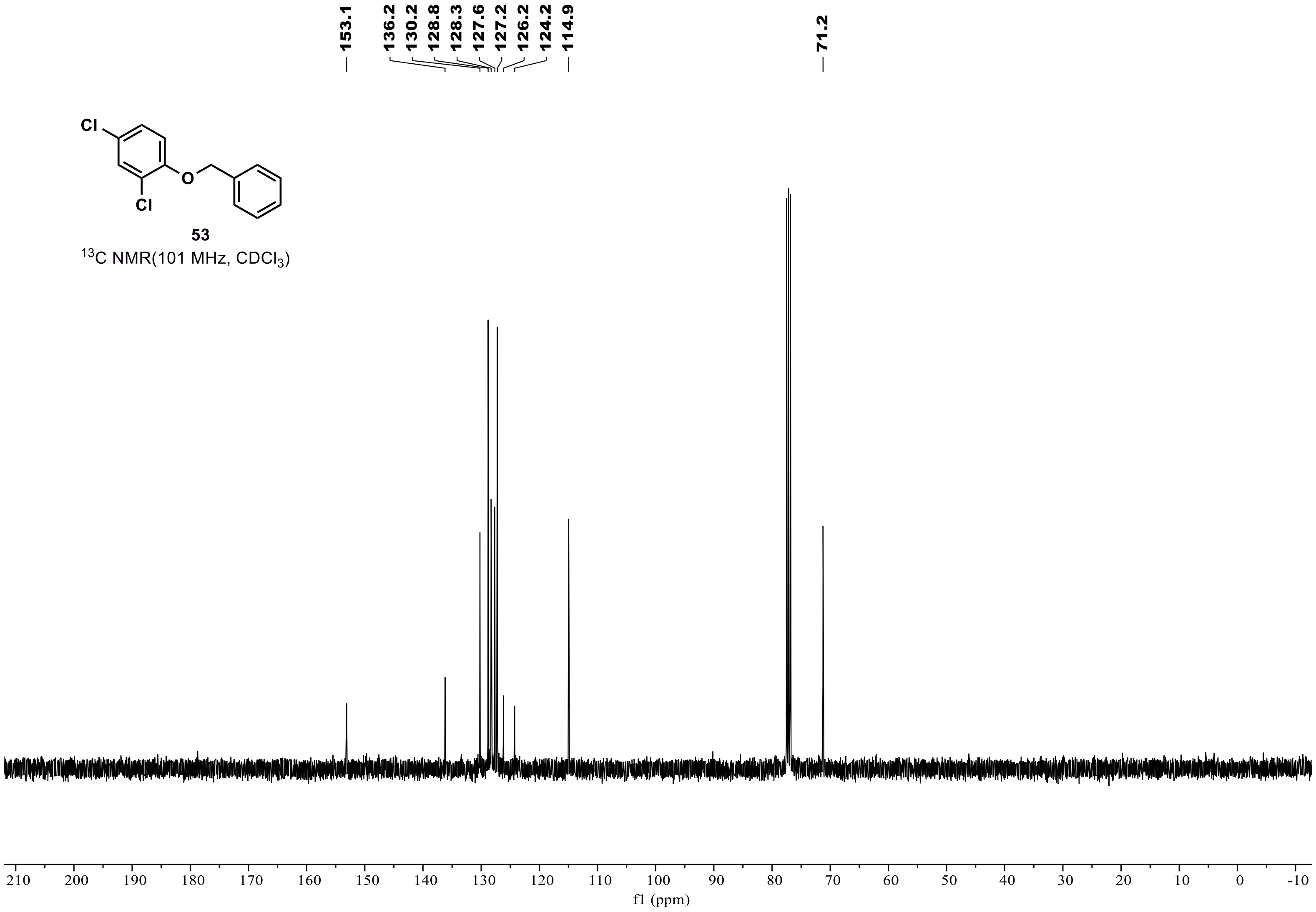
52

^{13}C NMR(101 MHz, $CDCl_3$)



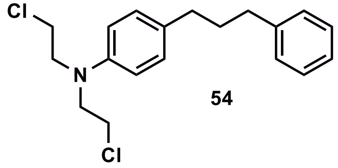
53

^{1}H NMR(400 MHz, $CDCl_3$)



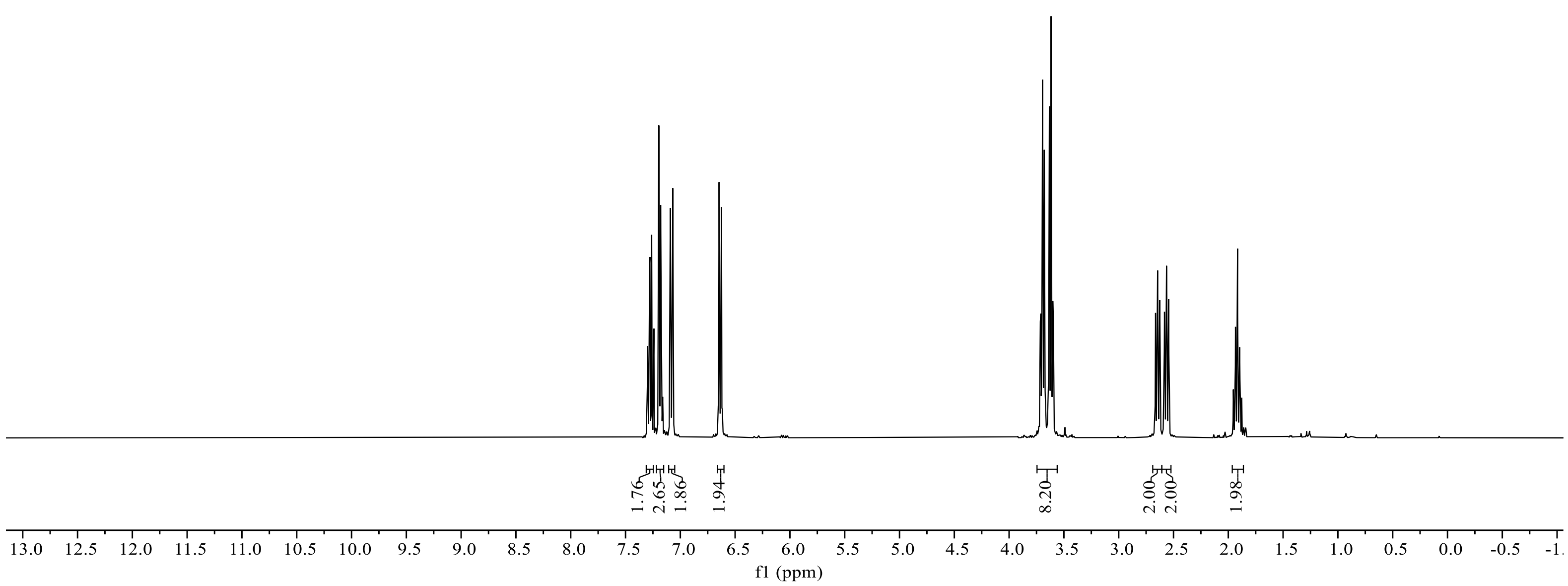
53

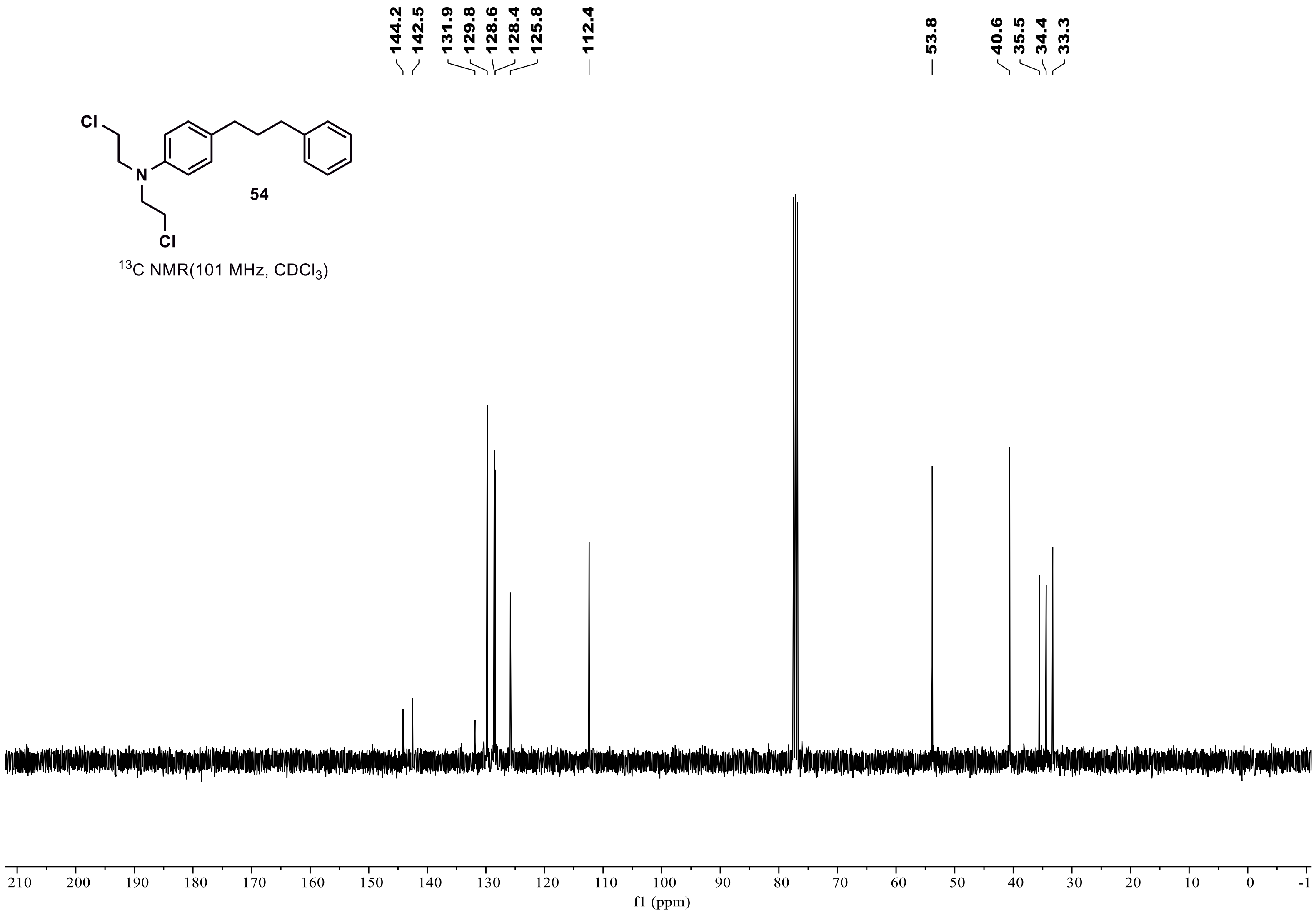
^{13}C NMR(101 MHz, $CDCl_3$)



54

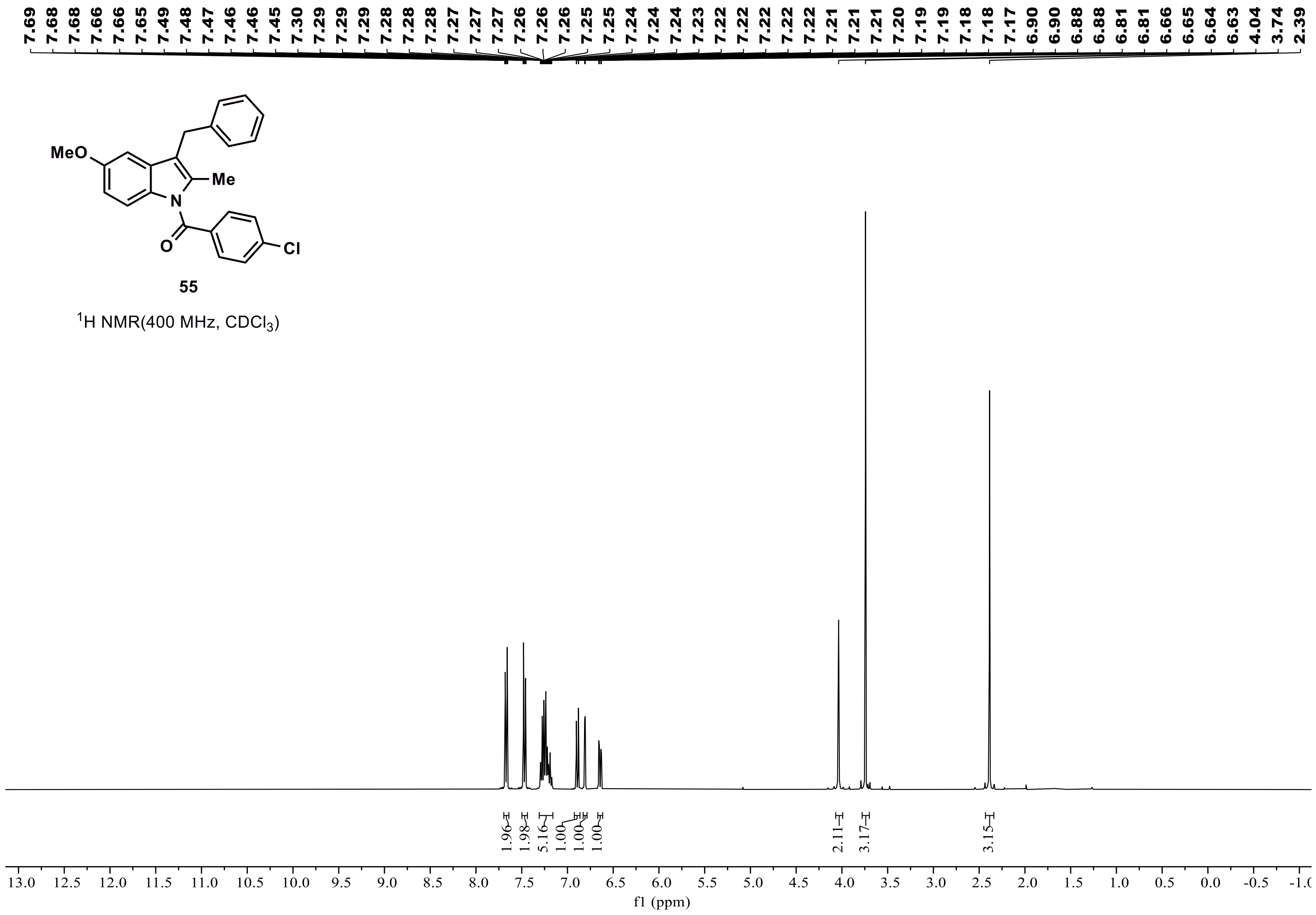
^1H NMR(400 MHz, $CDCl_3$)





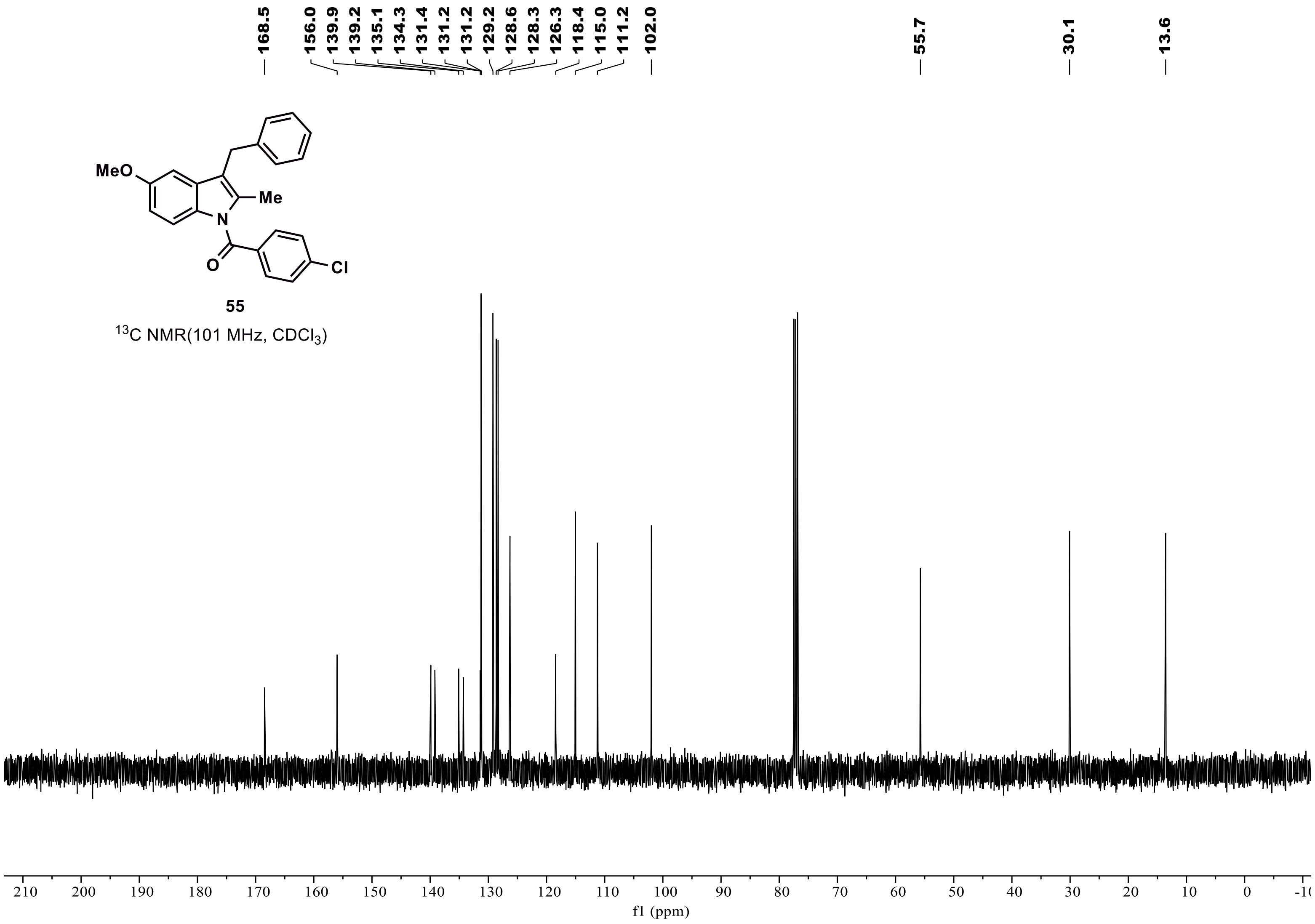
54

^{13}C NMR(101 MHz, CDCl$_3$)



55

¹H NMR(400 MHz, $CDCl_3$)



55

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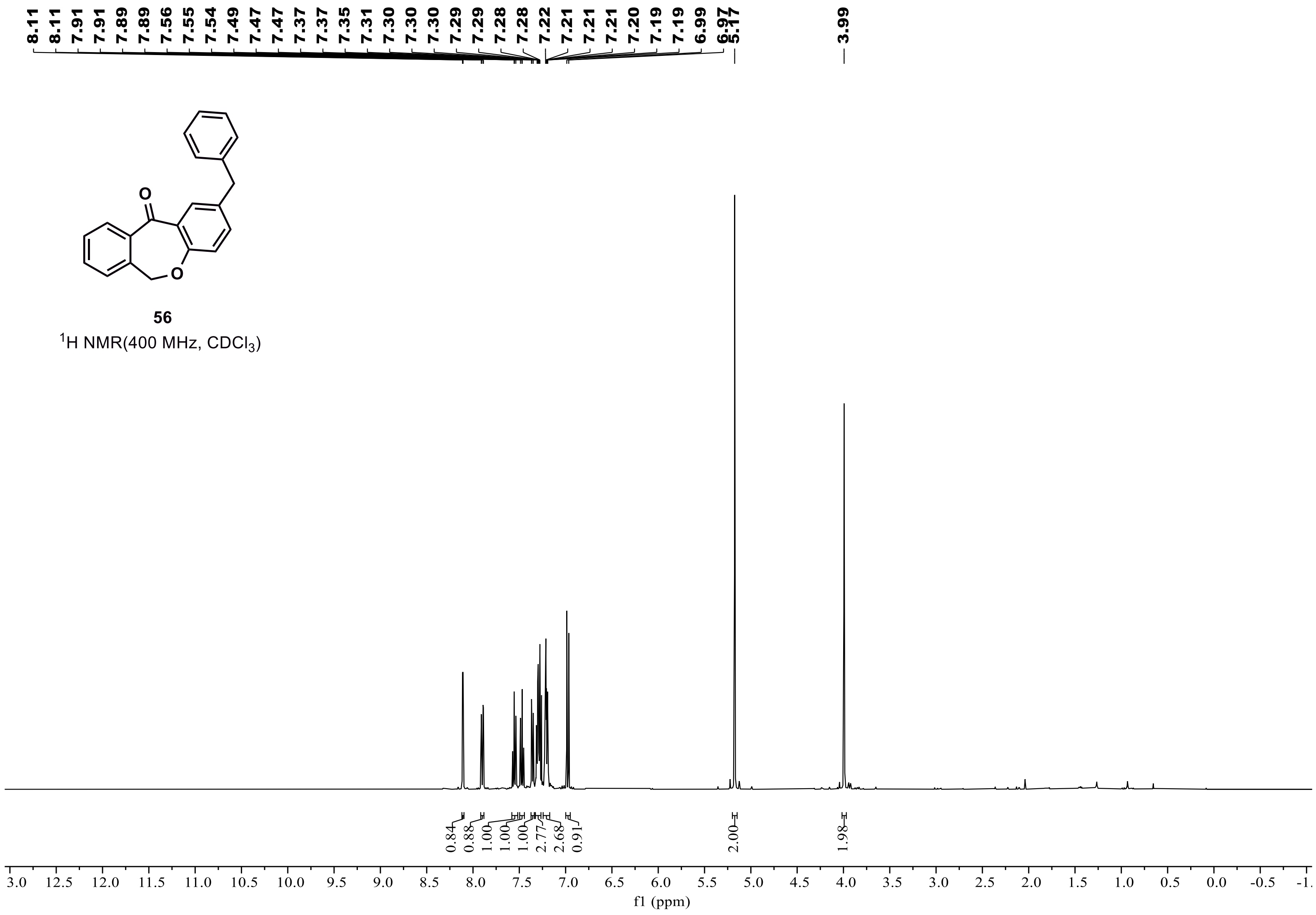
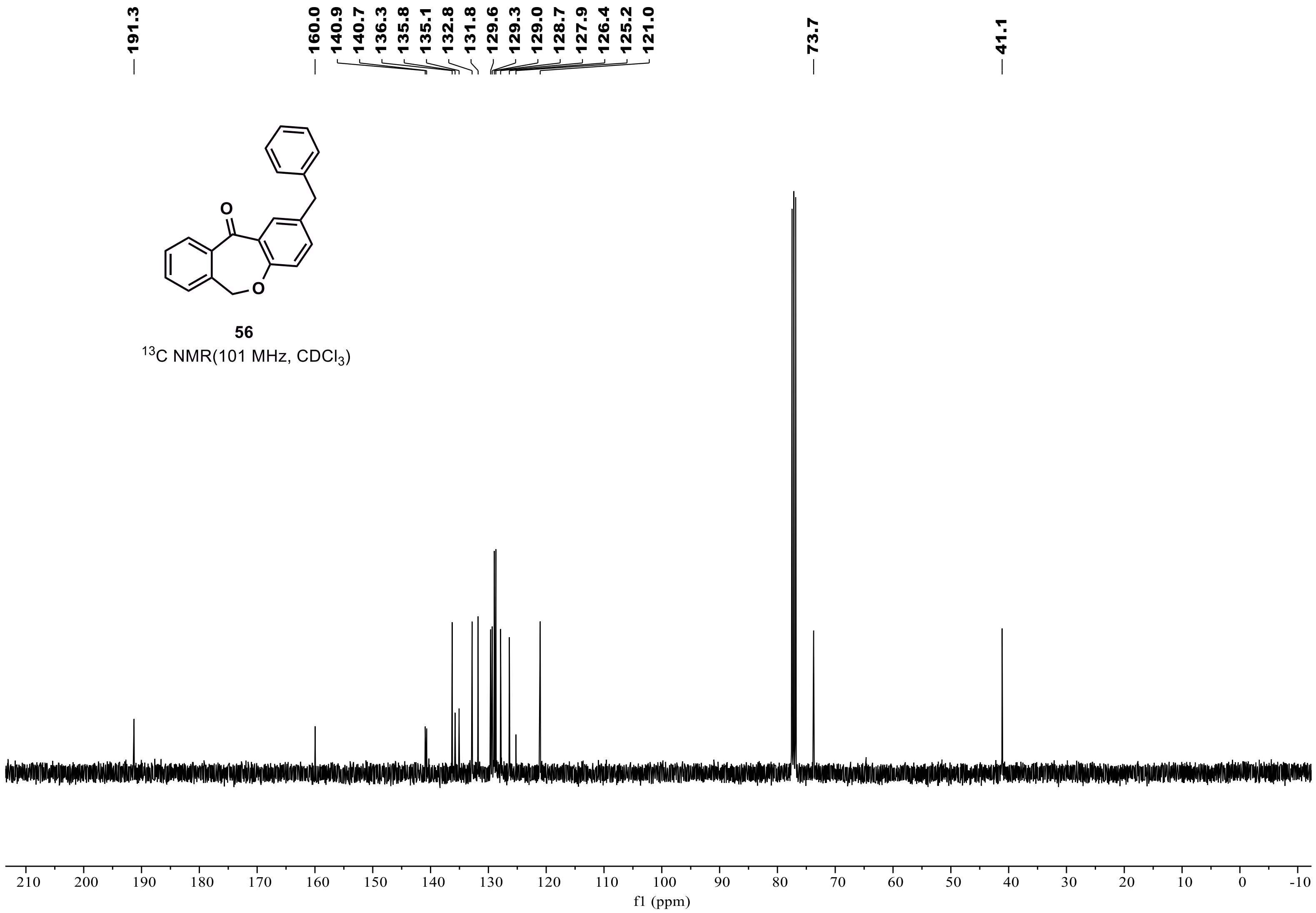


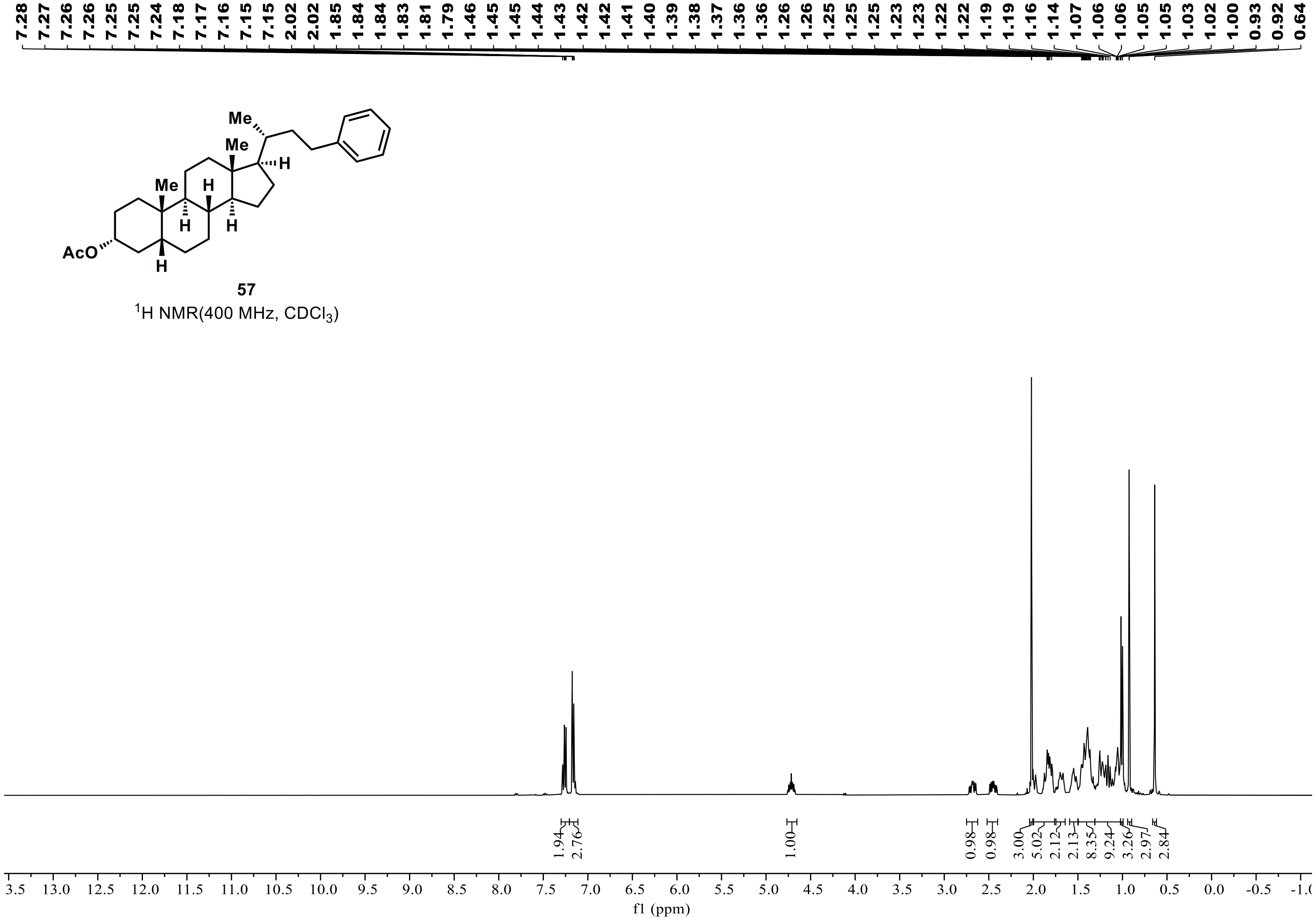
56

^{1}H NMR(400 MHz, CDCl$_3$)



56

^{13}C NMR(101 MHz, CDCl$_3$)



57

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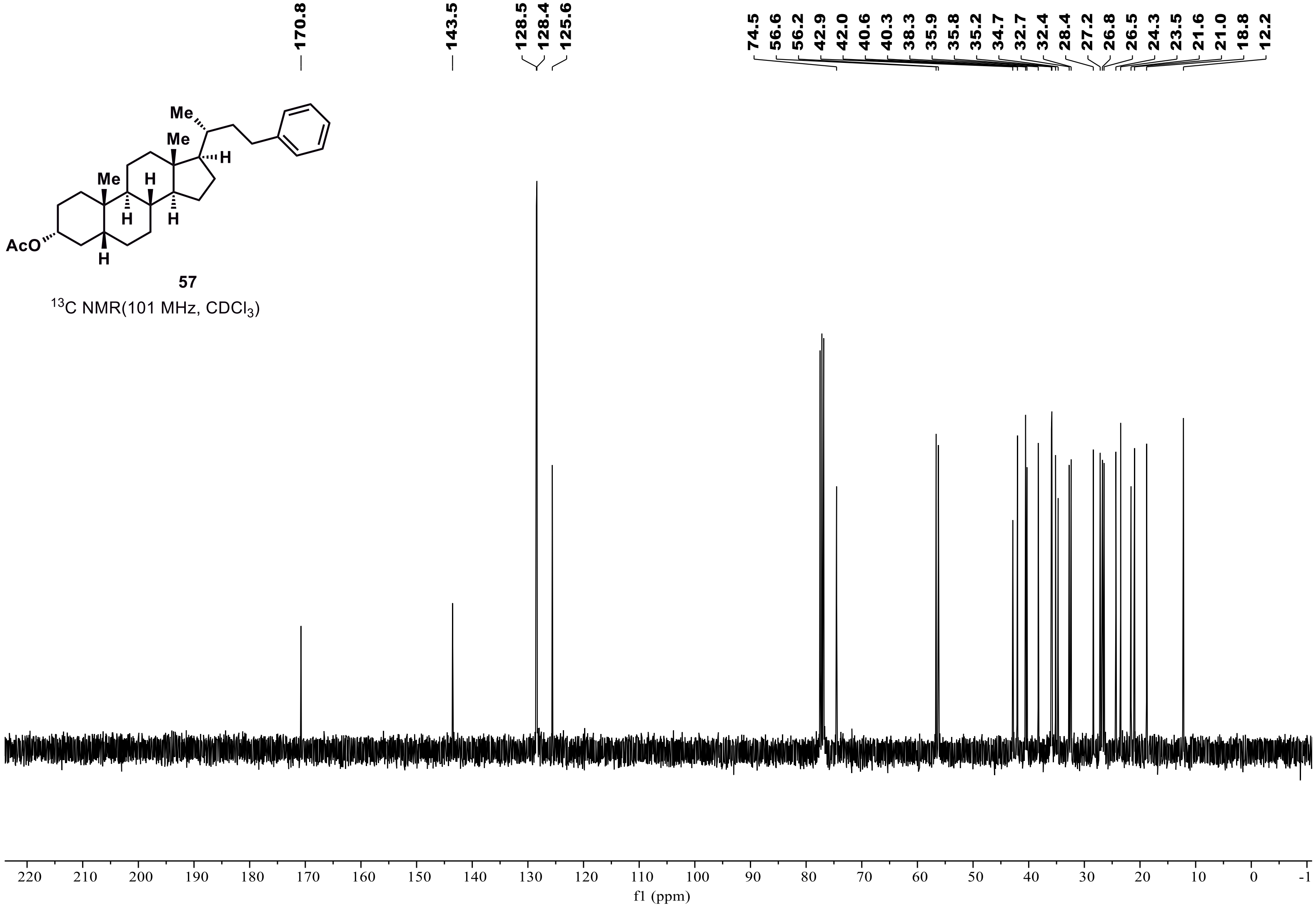
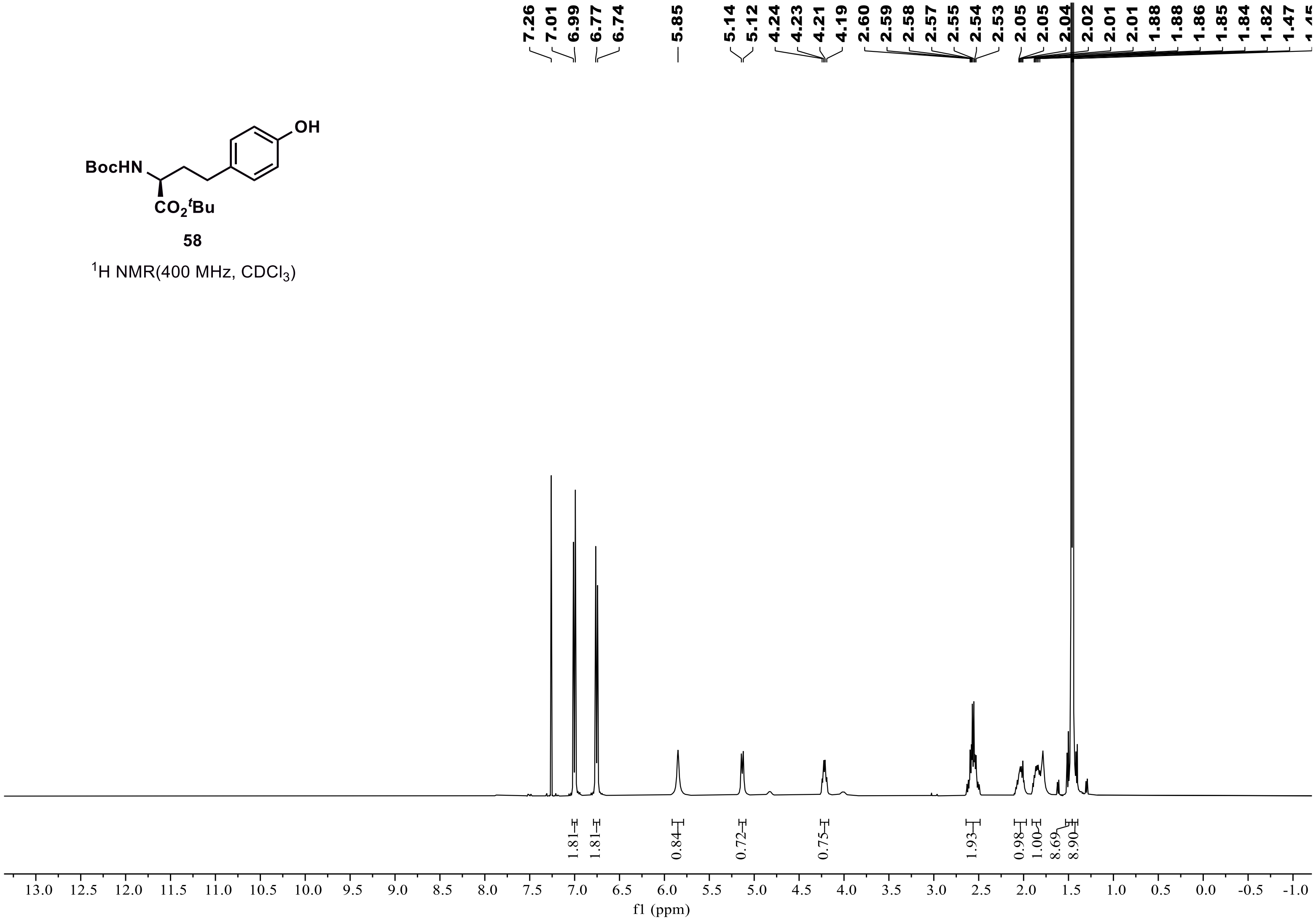


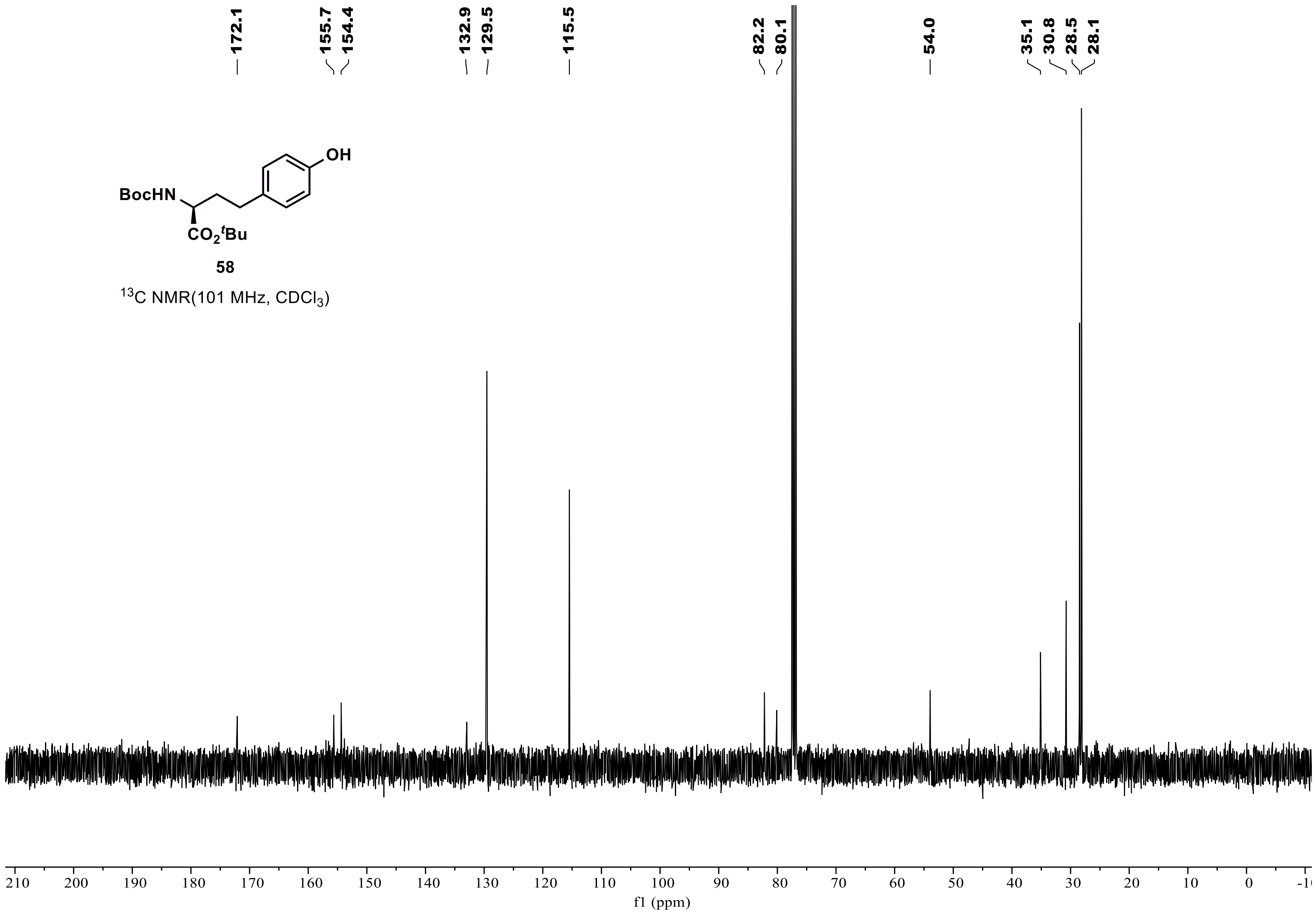
57

^{13}C NMR(101 MHz, CDCl$_3$)



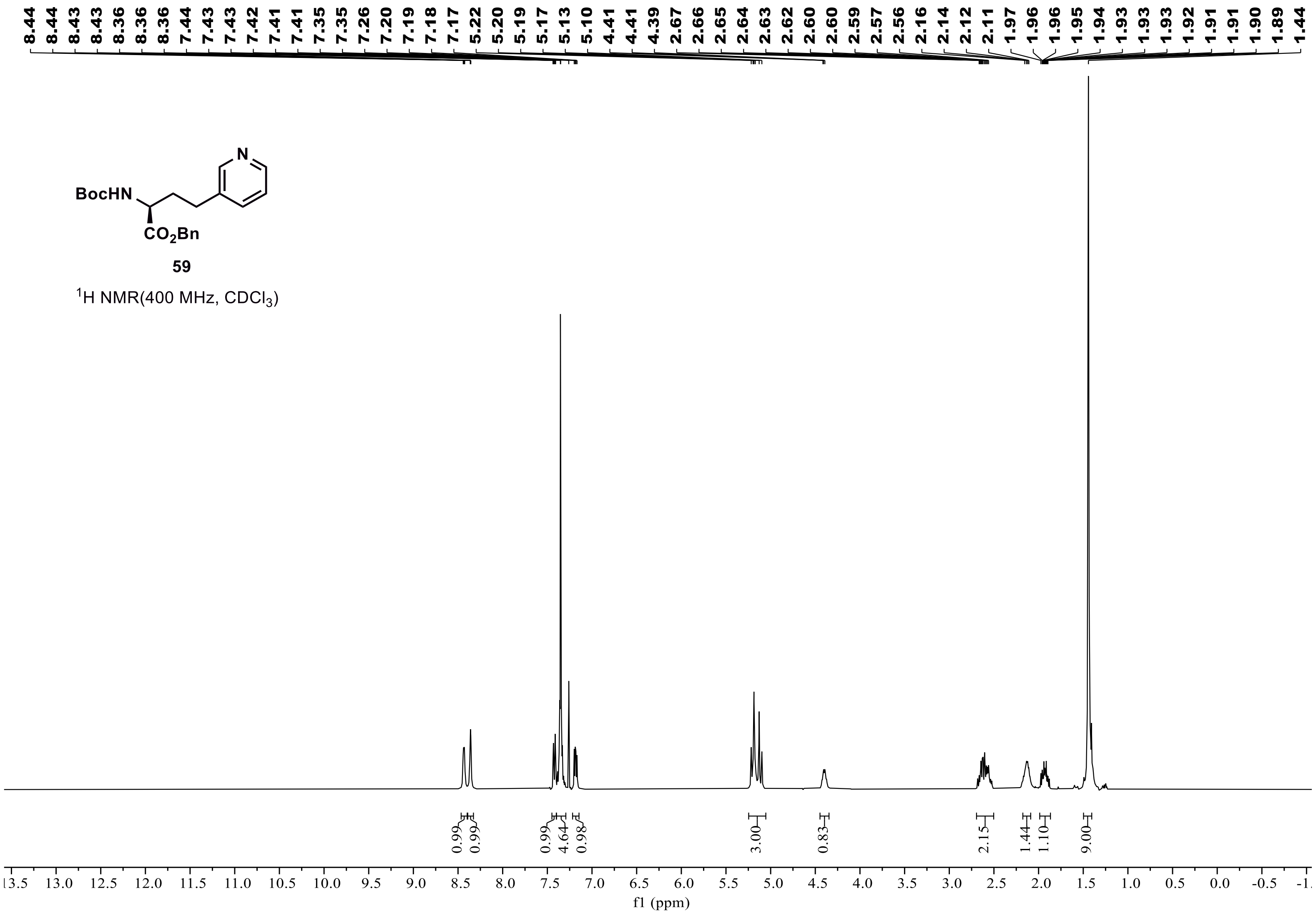
58

^1H NMR(400 MHz, $CDCl_3$)



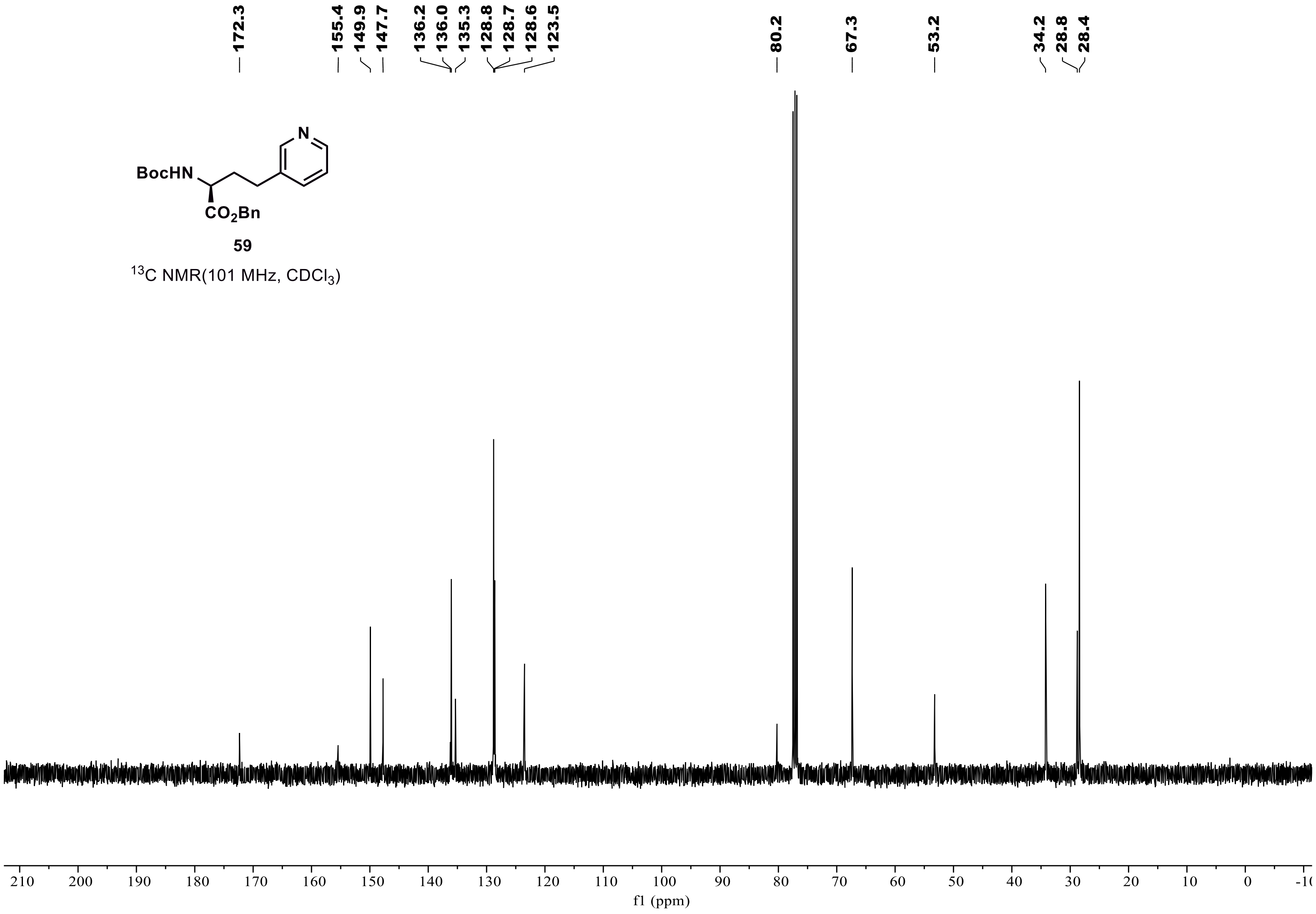
58

^{13}C NMR(101 MHz, $CDCl_3$)



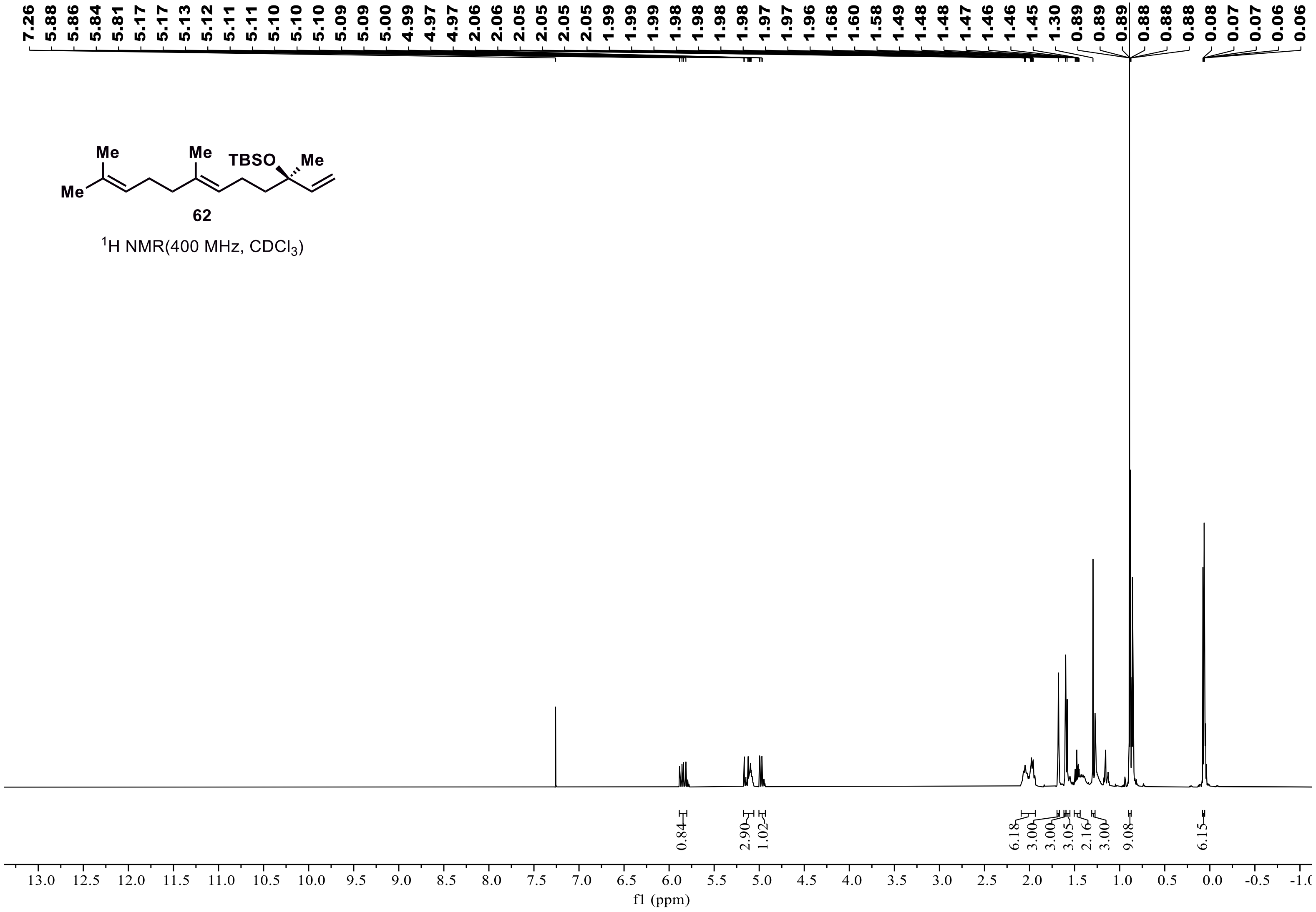
59

^{1}H NMR(400 MHz, CDCl$_3$)



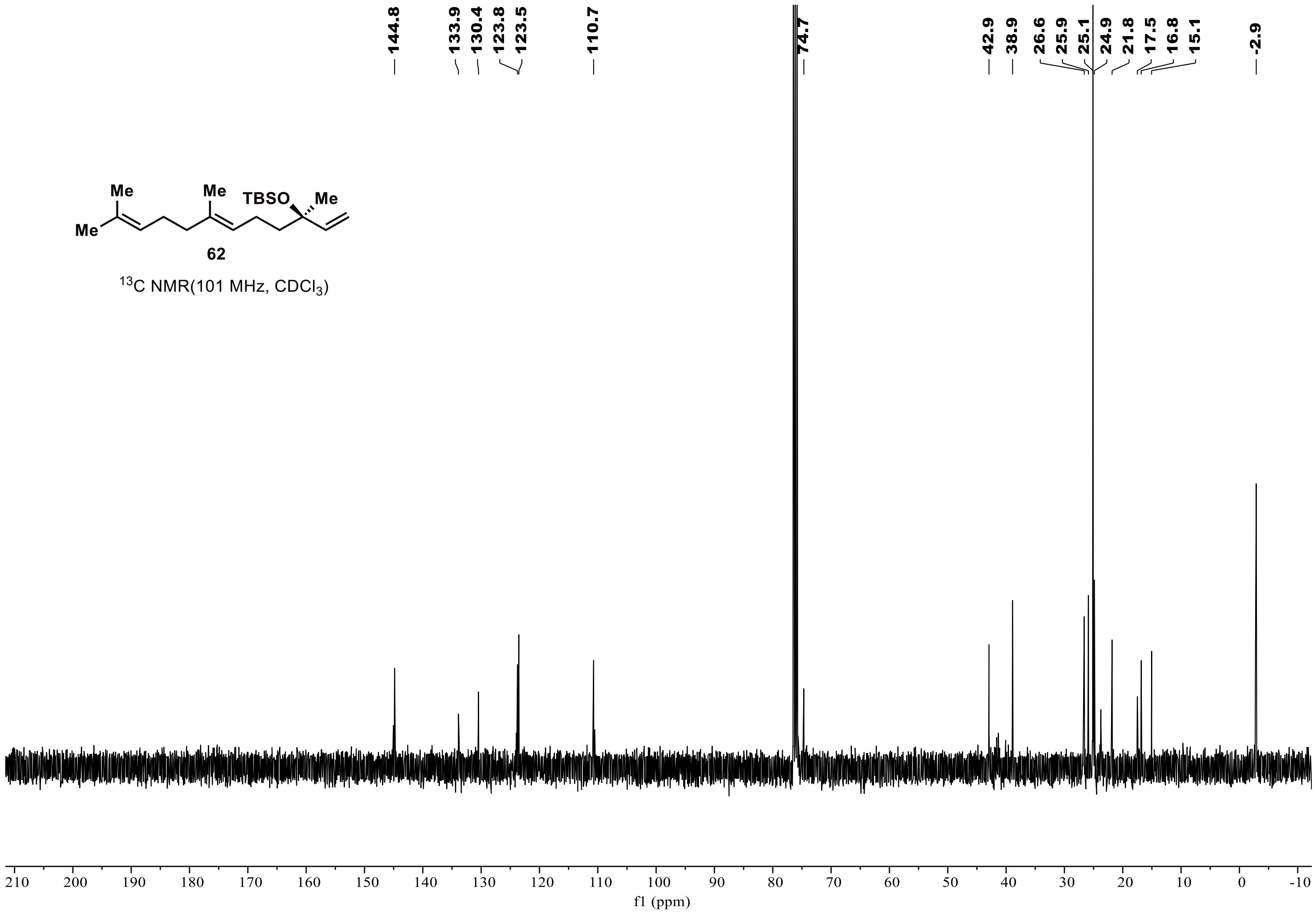
59

^{13}C NMR(101 MHz, CDCl$_3$)



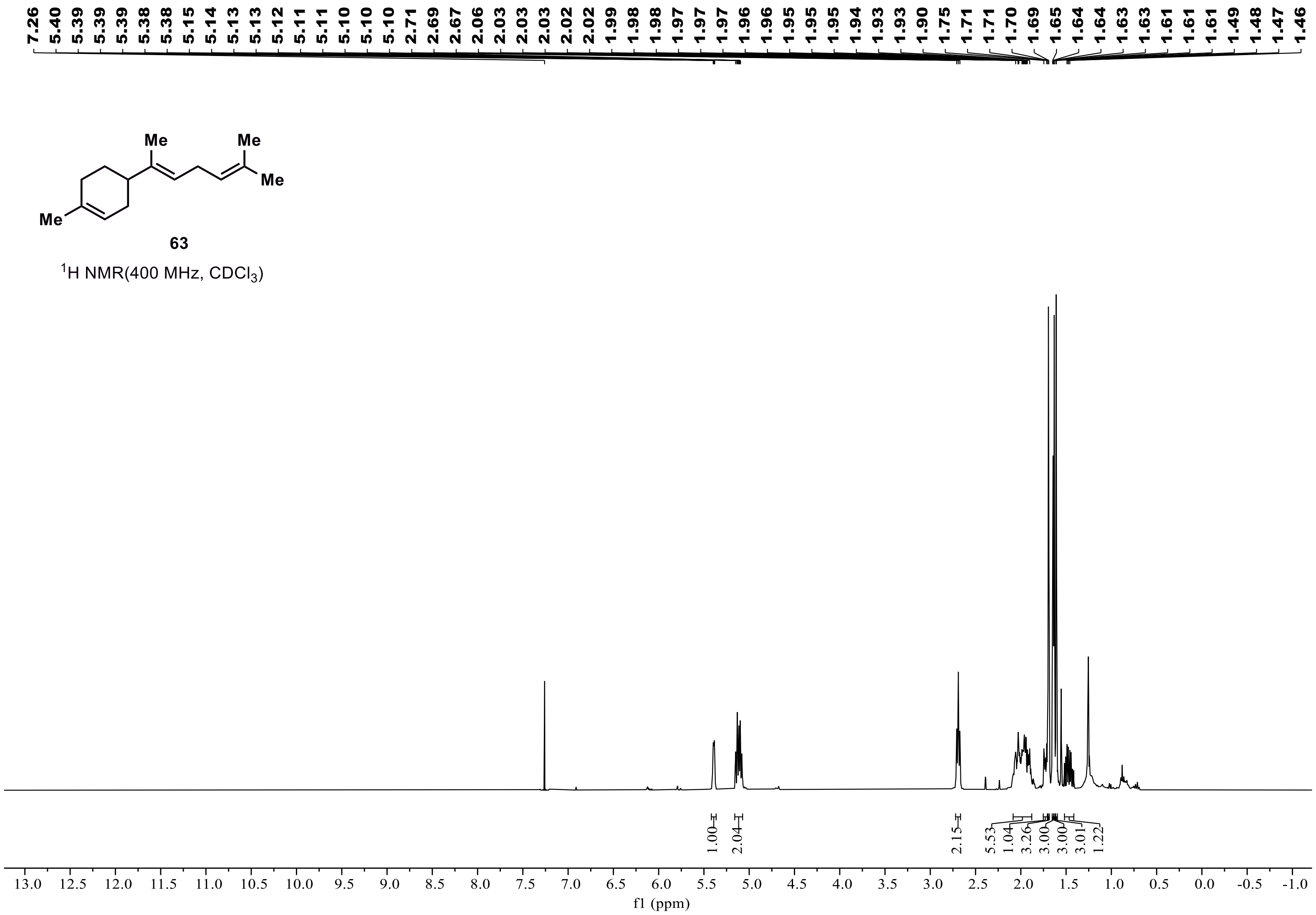
62

^{1}H NMR(400 MHz, CDCl$_3$)



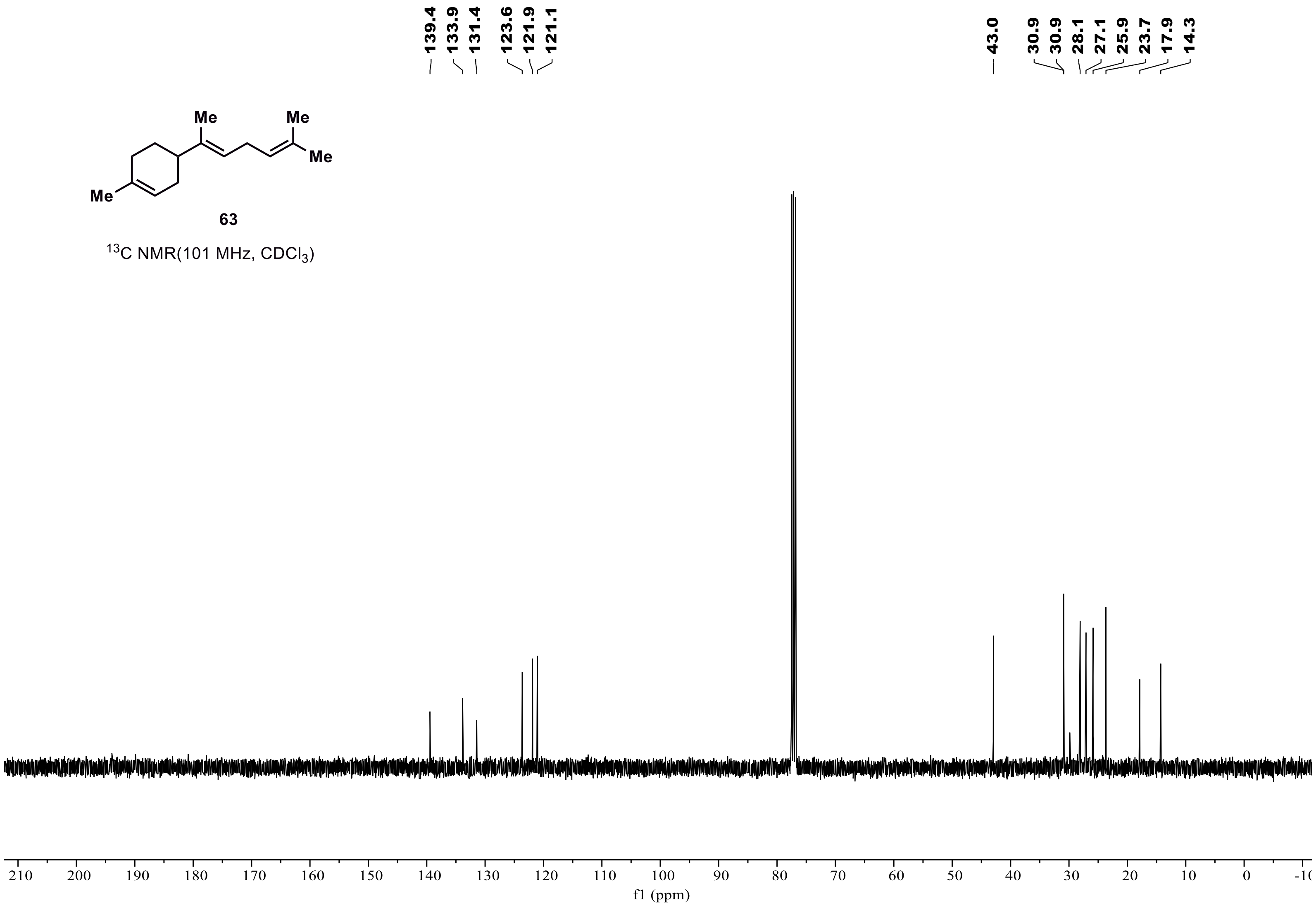
62

^{13}C NMR(101 MHz, CDCl$_3$)



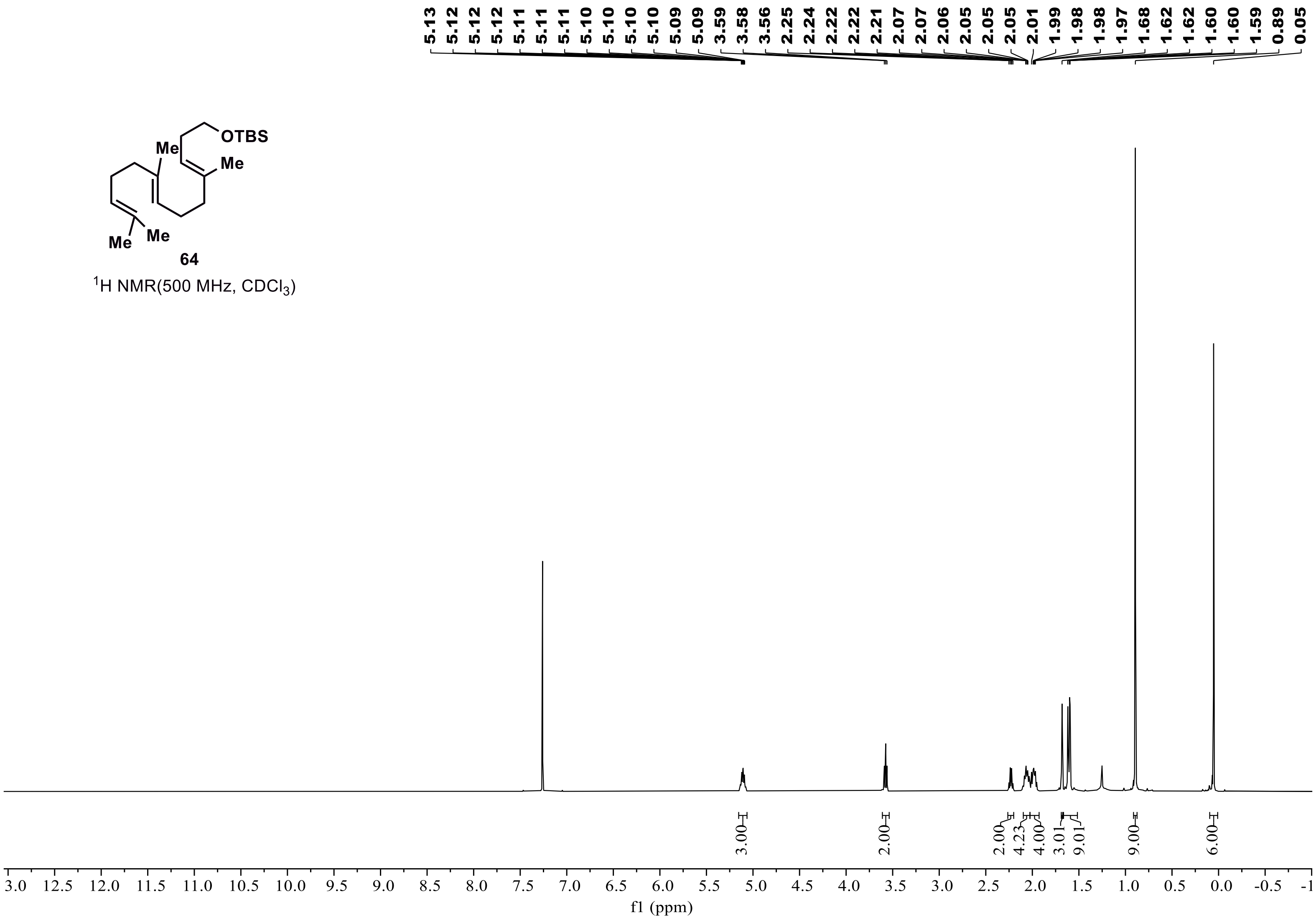
63

^{1}H NMR(400 MHz, $CDCl_3$)



63

^{13}C NMR(101 MHz, $CDCl_3$)



64

^{1}H NMR(500 MHz, CDCl$_3$)

64

^{13}C NMR(101 MHz, CDCl$_3$)